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American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS, PART II

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

SUMMARY OF PROCEEDINGS OF THE PITTSBURGH REGIONAL MEETING—SYMPOSIUM ON HIGH-STRENGTH CONSTRUCTIONAL METALS

The seventh regional meeting of the Society was held in Pittsburgh, Pa., at the Hotel William Penn on Wednesday, March 4, 1936, in conjunction with the Spring Group Meeting of A.S.T.M. Committees. Arrangements for the regional meeting were under the auspices of the Pittsburgh District Committee.

The morning and afternoon sessions of the meeting comprised a technical program, a Symposium on High-Strength Constructional Metals. The committee immediately in charge of the technical program consisted of the following:

Jerome Strauss, (*Chairman*), Vice-President, Vanadium Corporation of America.
F. H. Frankland, Chief Engineer, American Institute of Steel Construction.
R. A. Wilkins, Vice-President, Revere Copper and Brass Incorporated.
E. H. Dix, Jr., Chief Metallurgist, Aluminum Research Laboratories, Aluminum Company of America.
Dean Harvey, Materials Engineer, Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co.

The papers cover chemical and physical properties and manufacturing and fabricating properties of metals and alloys employed for such constructional applications as buildings, bridges, ships, railway car bodies, automobile bodies, airplane wings, tanks, containers for the chemical industry, etc. The symposium consisted of the following 5 papers:

"Carbon and Low-Alloy Steels," by Edwin F. Cone, Editor, *Metals and Alloys*.
"Corrosion-Resisting Steels," by Ernest E. Thum, Editor, *Metal Progress*.
"Alloys of Copper," by C. H. Davis, Metallurgist, The American Brass Co.
"Alloys of Nickel," by G. F. Geiger, Research and Development Dept., The International Nickel Co.
"Alloys of Aluminum and Magnesium," by E. H. Dix, Jr., Chief Metallurgist, and J. J. Bowman, Metallurgical Division, Aluminum Research Laboratories.

The co-chairmen of the sessions at the symposium were:

Morning Session:

N. L. Mochel, Metallurgical Engineer, Westinghouse Electric and Manufacturing Co.
R. F. Mehl, Director, Metals Research Laboratory, and Head, Department of Metallurgy, Carnegie Institute of Technology.

Afternoon Session:

H. W. Gillett, Metallurgist, Battelle Memorial Institute.
H. F. Moore, Professor of Engineering Materials, University of Illinois.

The papers elicited a considerable amount of discussion which contributed greatly to the information contained in the papers. The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on High-Strength Constructional Metals."

There were approximately 225 members and guests in attendance at the sessions.

The evening session, the Regional Meeting Dinner, which was of an informal nature, included talks by Dean Harvey, Chairman of the Pittsburgh District Committee, on "The A.S.T.M. District Committee Plan"; B. H. Witherspoon, President, Pittsburgh Testing Laboratories, on "The Role of the Commercial Testing Laboratory"; President H. S. Vassar on "Recent Developments in the Society"; and finally the principal address of the evening by S. M. Kintner, Vice-President in Charge of Engineering, Westinghouse Electric and Manufacturing Co., on the subject, "Electricity Marches On." This was followed by two reels of a sound film entitled "The New Frontiers," shown through the courtesy of the Westinghouse organization. Dr. Paul D. Foote, Director, Gulf Research Laboratory, The Gulf Companies, was toastmaster at the dinner.

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON RADIOGRAPHY AND X-RAY DIFFRACTION METHODS

The Symposium on Radiography and X-ray Diffraction Methods was held at the Thirty-ninth Annual Meeting of the American Society for Testing Materials at Atlantic City, N. J., on June 30 and July 1, 1936. Four sessions were held, two on the afternoon and evening of June 30, devoted to Radiography, and the other two on the morning and afternoon of July 1, devoted to X-ray Diffraction Methods. The chairman of the Radiography sessions was H. H. Lester, with R. H. Aborn, W. C. Hamilton and C. A. Adams as co-chairmen of the first session; and John T. Norton, C. W. Briggs and R. A. Bull as co-chairmen of the second session. The first Diffraction session was under the co-chairmenship of William L. Fink and Eric R. Jette; and the second Diffraction session was under the co-chairmenship of R. M. Bozorth and J. D. Hanawalt.

The program was sponsored and developed by Subcommittee VI on X-ray Methods, of the Society's Committee E-4 on Metallography. Mr. R. F. Mehl, chairman of Subcommittee VI, served as chairman of the Symposium Committee. The program covered two major phases of the the subject: first, Radiography, comprising six papers; and second, X-ray Diffraction Methods, also comprising six papers. The complete list of papers follows:

Afternoon and Evening Sessions—June 30

Radiography

"The Principles of the Radiographic Process," by John T. Norton, Associate Professor of Metallurgy, Massachusetts Institute of Technology.

"Foundry Applications of Radiography," by Earnshaw Cook, Research Metallurgist, Metallurgical Dept., The American Brake Shoe and Foundry Co.

"Radiography in the Welding Shop," by J. C. Hodge, Chief Metallurgist, The Babcock & Wilcox Co.

"Miscellaneous Applications of Radiography and Fluoroscopy," by Herman E. Seemann, Physicist, Kodak Research Laboratories.

"Gamma-ray Radiography and Its Relation to X-ray Radiography," by Norman L. Mochel, Metallurgical Engineer, Westinghouse Electric and Manufacturing Co.

"The Problem of Radiographic Inspection," by H. H. Lester, Senior Physicist, Watertown Arsenal.

Morning and Afternoon Sessions—July 1

X-ray Diffraction Methods

"X-ray Diffraction Equipment and Methods," by Charles S. Barrett, Member of the Staff of the Metals Research Laboratory, and Lecturer, Department of Metallurgy, Carnegie Institute of Technology.

"The Constitution of Alloys," by Kent R. Van Horn, Research Metallurgist, Metallurgical Division, Aluminum Research Laboratories.

"Chemical Analysis by X-ray Diffraction Methods," Wheeler P. Davey, Research Professor of Physics and Chemistry, School of Chemistry and Physics, Pennsylvania State College.

"Applications of X-ray Methods to Problems of Cold Work, Preferred Orientation, and Recrystallization," by John T. Norton, Associate Professor of Metallurgy, Massachusetts Institute of Technology.

"The X-ray Determination of Particle Size," by G. Harvey Cameron, Professor of Physics, Hamilton College, and A. L. Patterson, formerly Department of Physics, Massachusetts Institute of Technology. Present address, Department of Physics, Bryn Mawr College, Bryn Mawr, Pa.

"The Applications of the X-ray Diffraction Method to Non-Metallic Materials," by George L. Clark, Professor of Chemistry, Department of Chemistry, University of Illinois, Urbana, Ill.

The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on Radiography and X-ray Diffraction Methods."

DEVELOPING AMERICAN GLASS

Edgar Marburg Lecture[1]

By Arthur L. Day[2]

Synopsis

American glass development was slower in starting than the corresponding European development, but since its beginning, early in the present century, it has been more intensive and in many fields has proceeded farther, notably in the application of machinery to forming processes. Most of the glass-forming machinery in use in the world today is American made. Historically, it is within the memory of men now living that the glassmaker's salesmanship consisted in convincing purchasers that his particular glass was better for all purposes than any other. Today, leading glassmakers offer several hundred compositions, each adapted to a particular purpose and often limited to that purpose, and the number of such purposes increases daily. Of almost equal importance is the recent development of physical tempering of glasses through which a given composition may be made to serve a wide variety of purposes with success.

Conspicuously American is the development of large-scale continuous-melting units for the production of low-cost glass in quantity. Then follow the mechanical feeding devices through which a continuous supply of molten glass is distributed to forming machines (for containers, electric light bulbs, etc.), drawing machines (for tubing, window glass, etc.), presses and the like. Practically all of these machines are now fully automatic and capable of adaptation to almost any need of present-day quantity production.

Developments of the last decade have shown that the usefulness of glass is not limited to forms which can be annealed and freed from physical strain, indeed if the strain is adequately controlled and properly oriented, it may increase immensely the physical strength of glass and, so, add to the number of its social uses.

Among the advances in chemical composition the transition from soda-lime-silica glass, which dominated all fields of glass-making activity from early Egyptian times to the beginning of the present century, to the great group of borosilicate glasses which show extraordinary stability of composition, resistance to weathering or chemical attack and adaptability to manifold forming operations, is outstanding. Of these glasses the latest and greatest is the 200-in. telescope disk successfully cast for the California Institute of Technology with an expansion coefficient only one-fourth as great as window glass.

John Masefield has somewhere said that an expert is always dull, for he knows only his job and nothing of its philosophy. It is therefore no part of my purpose to qualify as an expert. I would wish to go back to a viewpoint from which a broader perspective, philosophical if you like, may be brought to bear upon the development of the glass industry in this country, and perhaps to pass in somewhat closer review the vigorous research trend developed within the present century.

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[1] Read on July 1, 1936, before the Annual Meeting of the American Society for Testing Materials, Atlantic City, N. J.

[2] Director, Geophysical Laboratory, Carnegie Institution of Washington, Washington, D. C.

Glass itself is as old as the earth. It is a natural and not a man-made product, originally. The stone-age men had it and there are to be seen in many museums the arrowheads, spearheads, axes and the like which have documented the earliest acquaintance of primitive man with glass. In this country glass artifacts are still found near the places where volcanic obsidian, as the natural glass is called, is available.

In the Yellowstone National Park there is a mass of obsidian about nine miles long, five miles wide, and rising about 250 ft. above the level of the adjacent country, which is, throughout, a massive, black glass. A number of years ago when the plan for the great 200-in. disk, which has been so much in the public press during the last few weeks, was first conceived, Doctor Hale had the definite idea that if such a disk could not be made, it would still be possible to go to the Yellowstone Park, quarry out a section of the obsidian and use it for a great reflecting telescope. It was not attempted; perhaps it would have proved to be a difficult and expensive task to find a 17-ft. mass free from surface flaws. Furthermore, such a disk would have had an expansion coefficient much too high for Doctor Hale's purpose, so perhaps it is just as well that the attempt was not made. It is nevertheless a valuable native product which still has its uses.

Man-made glass began to be made about five thousand years ago; that is to say, the oldest glass fragment in the Department of Oriental Art at the University of Chicago dates from about 3000 B. C. and had its origin in Asia Minor. In the eighteenth Egyptian dynasty, or about fifteen hundred years before Christ, the glass industry flourished, and from then on to the time of the greatness of Rome, glassmakers were common enough. The glass of those days, however, was not used for the purposes that we associate with it today. For the most part it appears to have been used for ornament or to provide small containers for treasured perfumes; but glass to be used as a transparent medium had not, up to that time, been made. All of the glasses of the earliest period were more or less colored.

The beginning of glass-making in this country dates from 1608, only a year after the landing of the first Jamestown colonists. I suppose the claim may be made that it is the oldest American industry of which we have a record. The Jamestown colonists brought it with them, ready-made, from England and set it up immediately in Virginia for the purpose of making bright beads which might be traded profitably with the Indians. Indeed for a considerable while after that, all of the output of this glass-making group was designed for ornament. The glass needed for windows was imported from England for at least a century thereafter.

There is one curious thing which should be emphasized, namely, that the composition of all of these glasses from the earliest sample we have, down to the glass bottle of today's vintage, is substantially the same; the differences are insignificant from a chemical standpoint, and the reason for that will presently appear in the course of this discussion.

Man-made glass can hardly have originated in attempts to imitate a natural product. It would not have been possible for the ancient glassmaker to imitate the natural glass (obsidian). It has a melting temperature much too high to have been liquefied and shaped in the early fires that were available for such an undertaking. So that I think it altogether improbable that the early glassmakers ever attempted to imitate natural glass by artificial means. The natural glass is a mixture of feldspar and silica and would hardly have a softening point below 1400 C. or something of that order of magnitude, and even there its viscosity would be too great to permit fabrication into predetermined forms.

The beginnings of window glazing are obscure but it may be presumed that the wonderful cathedral windows which served somewhat literally to illuminate the dark ages were preceded by cruder efforts to admit light into dark interiors of which the record is now lost in the shadows of that early period. Certain it is that the glory of the glass-making art has centered in Venice from the time of the Roman Empire almost to the present and the Venetian craftsman has left many a monument to his high artistic ideals which has not been surpassed in any age or country before or since.

Considering glass development logically rather than historically, I suppose the simplest type of glass that can be made artificially is pure molten silica, although we think of this as a fairly modern product. Ordinary sand, if you have heat enough to melt it, makes a very creditable glass indeed, so good that if it could be easily manufactured at accessible temperatures, it would perhaps serve all of our purposes as well as or better than most of the glasses available today for containers or for engineering purposes. The glass has a very low expansion coefficient, about one-tenth that of ordinary window glass; it is transparent to all of the wave lengths of the visible spectrum and also to the ultraviolet, which happens to be the main purpose for which it is now used, but it is exceedingly difficult to reduce to a homogeneous melt. Although sand is common enough, is uniform in texture and in its chemical composition, and is cheap, nevertheless the melting temperature of the high crystal form of silica is 1713 C., which is quite out of the reach of ordinary commercial furnaces. Of course, if sufficient heat is applied to silica it will melt, but the immediate product is not a clear glass suitable for a window or for a bottle, and the reason for that is inherent in the properties of the material itself.

Glass, silica glass, is made by heating quartz sand at appropriate temperatures; at about 575 C. quartz changes its crystal form to another which occupies several per cent greater volume—which means that the original fragments will disintegrate at that point admitting air freely between the grains. If heating is continued until melting occurs, the effect will be to include in the viscous mass a multitude of bubbles of air, large and small, forming an opaque pumiceous mixture the viscosity of which, if you are fond of large figures, runs to several million poises. The viscosity of molten quartz is such that no method is known, at the moment, of getting out or keeping out the bubbles of air.

I experimented with quartz melting myself a number of years ago and published an article on the subject in *Science* as early as 1906.[3] The ingenuity which we applied to the problem, or endeavored to apply, in order to obtain clear silica glass, was probably worthy of a better cause, as it has since turned out. Nevertheless we did obtain creditable glass by the following somewhat difficult means:

We built an electric melting unit which could be evacuated, pumped it down to as good a vacuum as we could establish in an electric furnace with graphite electrodes passing through the end walls (a few millimeters of mercury) and melted the quartz in that attenuated atmosphere. Then before any opportunity had been given for the molten mass to harden, that is to say before the temperature was lowered, the vacuum was replaced with about 600 lb. of air pressure, the thought being to eliminate as much of the air from between the grains as possible before and during melting, and to reduce the volume of the remainder, trapped as bubbles, by applying pressure to the fluid silica. The method yielded blocks of glass of considerable size, of reasonably good appearance and transparency, but there were some residual bubbles, all small. There were also shadowy, superficial discolorations due to the fact that at that temperature, in contact with graphite, a little silicon had been set free; but generally speaking, the method proved practical, within limits, and it is substantially the one which was used on a larger scale at Lynn, during and after the War, by Prof. Elihu Thomson.

Professor Thomson, in his experience, did not escape from the limitations and difficulties inherent in the properties of molten silica which confronted me twenty years before; in fact, an officer of his company once said to me across a dinner table, "Didn't you, in earlier years, experiment with quartz glass?" And when I said "Yes," he asked "How did you know when to stop? We have made silica glass successfully at Lynn but at a cost nearly equal to its weight in gold and there is no market for quartz glass at the price of gold";—which pretty well describes the situation today.

Of course quartz glass, if we had it, would be practically fireproof; I mean that no ordinary fire would destroy a low-expansion glass of that strength. It is in fact stronger than the glasses we commonly use today. In this connection there comes to my mind a tale of long ago. A short while after we had published our early experiments with quartz glass, a man in Southern Virginia wrote to suggest that there might be mutual profit for him and for me if we would consider making silica-glass coffins which, he said, ought to be hellproof.

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[3] Arthur L. Day and E. S. Shepherd, "Quartz Glass," *Science* N. S. 23, April 27, 1906, p. 670.

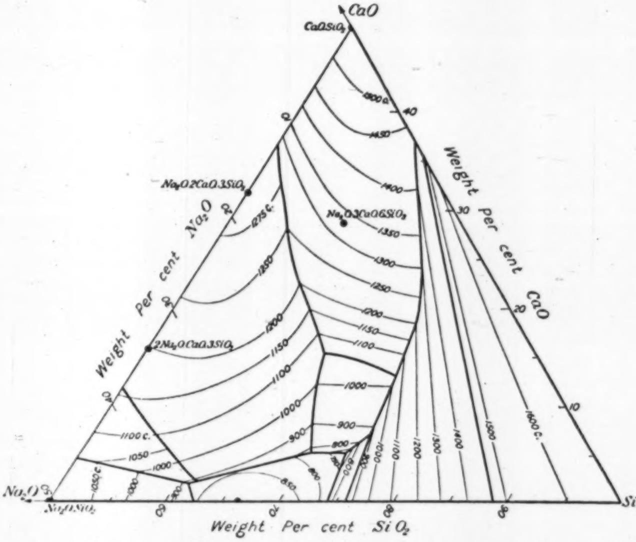

FIG. 1.—Measured Freezing Temperatures of Mixtures of Soda, Lime and Silica.

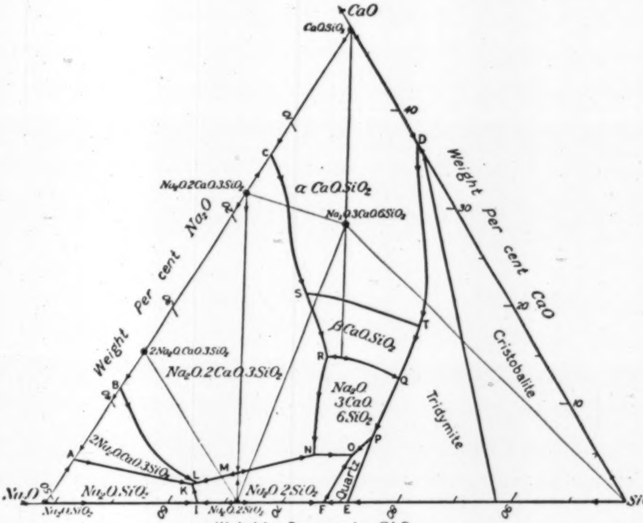
CaO
CaO.SiO₂
α CaO.SiO₂
β CaO.SiO₂
Na₂O.2CaO.3SiO₂
Na₂O.3CaO.6SiO₂
Na₂O 3CaO 6SiO₂
2Na₂O.CaO.3SiO₂
Na₂O.SiO₂
Na₂O.2SiO₂
Na₂O
SiO₂
Quartz
Tridymite
Cristobalite
A
B
C
D
S
T
R
Q
P
O
N
M
L
K
I
F
E
Weight Per cent Na₂O
Weight Per cent CaO
Weight Per cent SiO₂
40
30
20
10
80
70
60

FIG. 2—Compounds Formed Between Soda, Lime and Silica and Their Boundary Curve.

Now, starting from silica as the simplest glass both chemically and physically, extraordinarily stable and refractory when cold but somewhat difficult to make and hardly available to commerce because of its high cost, the first thought is to find a substance which will flux with it, which will make it melt easier, flow more freely and at a lower temperature, and so, bring it into such a condition that it can be worked into convenient forms at convenient temperatures. The most obvious step is to add a little alkali (potash or soda); about 26 per cent of alkali added to quartz reduces its melting point from 1700 to about 800 C. The resulting substance is familiar enough in various industries as water glass, but you would not think of using it for bottles or for windows. The limitation, of course, is that it is water-soluble, and to dispose of that, something more must be added. The cheapest material and the one, by the way, which the ancients used from the beginning, is lime. If lime be added, the water-solubility is gradually overcome and a resistant glass results. Transparency depends on the purity of the ingredients.

Soda-lime-silica is probably the simplest composition, after pure silica, from which we can obtain an industrial glass in which the viscosity can be varied with temperature so as to make the material workable, and which is transparent, which is of course in most cases a desirable quality.

So the early glasses and the late ones have adhered rather closely to a formula which contains these three ingredients, the lime, the soda and the silica. As the addition of the alkali reduced the silica melting point, so the addition of lime produces a further reduction, until finally the lowest temperature of the triple eutectic is reached at about 725 C. A diagram (Fig. 1) lately published by George W. Morey[4] of the Geophysical Laboratory, showing the measured freezing temperatures of mixtures of soda, lime and silica, is here offered to confirm to you the stubborn facts which have guided or driven both ancients and moderns, from somewhat different viewpoints to be sure, toward this composition because it is the lowest-melting, stable glass which is available to us. It is within the reach of such fires as may have been available to the ancients; it is also easier for us to work at a lower temperature than a higher one; furthermore, the viscosity of such a glass is appropriate for convenient working, and there is little tendency to devitrify at any temperature—above the melting point none at all.

To be serviceable in windows and in bottles, still more in the case of chemical ware, certain limitations must be imposed upon glass composition to insure stability and to limit solubility. Figure 2 is an equilibrium diagram, also published by Morey,[4] showing the compounds formed between soda, lime and silica and their boundary curves. I need not remind you that silicate mixtures in the vitreous state (glass) are never in equilibrium, but potential changes (with temperature) are so completely dominated by the viscosity that at the lower temperatures effective stability results.

Such considerations of chemical stability, or possibly a search for new optical properties, may have led Faraday to undertake to use a different flux with silica and so to reduce the amount of alkali in the glass composition if it should be possible to do so, and he was the first, I think, to try borax and boric acid for this purpose. The borosilicate glasses, at any rate, had their beginning in Faraday's laboratory about a century ago, although it was not until seventy years later, or thereabouts, that the borosilicate glasses came into general use. Most of us are entirely familiar with borosilicate glasses today; we have them in every chemical and physical laboratory in a wide variety of forms; we had them in war-time and earlier in our field glasses and fire-control instruments; we have them in "Pyrex" ovenware, and we use them daily in our photography. Latterly the outstanding application of borosilicate glass is in the 200-in. telescope disk which has found its way to California within the last few weeks. All of the glasses of the borosilicate group possess great stability both chemically and physically. Their expansion coefficient is only about one-third as great as window glass, and they may be subjected to all sorts of thermal abuse with little danger of breakage or devitrification. From the optical point of view, though we cannot go far in that direction, it has qualities also which are not found in other optical glasses. The borosilicate crowns were of the highest importance in equipping the army and navy with optical glass during the War.

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[4] George W. Morey, "The Composition of Glass," *The Scientific Monthly*, June 1936, Vol. 42 p. 547.

The stablest glass for nearly all purposes, then, is probably the borosilicate; it has a higher percentage of silica and a lower percentage of alkali than other glasses and has in consequence a higher melting or softening temperature, which is at the same time a disadvantage because it costs much more to melt it. The great 200-in. disk, which is of borosilicate composition, was melted at about 1550 C.; but after the glass is once melted its homogeneity is excellent, and, as already pointed out, its stability in the face of chemical attack and its physical resistance to breaking stresses, are very considerably higher than in other glasses.

Now if it is desired to impose upon glass other properties in addition to transparency, homogeneity and resistance to weathering, such as color in traffic-control signals, or special properties of light transmission, which are necessary in neon tubes, or any of the particular qualities useful for optical purposes, such as a definite refractive index or coefficient of dispersion, these qualities must be attained by the addition of other metallic oxides in carefully measured quantity or by a limited replacement of some part of our standard ingredients. There is no time for us to go into these widely developed specialties now, though they actually form one of the most interesting chapters in the development of American glass. For example, in a problem like that of the neon tube, where exposure to weather under widely different conditions of temperature and moisture must be confronted, it is obviously wiser to start out from a borosilicate glass for its initial stability. If it is then desired to superimpose upon that a quality of fluorescence such that the ultraviolet light in the discharge may become visible, this may be accomplished through fluorescent material incorporated in the glass itself or by means of a fluorescent coating outside or inside after the tube is made. These refinements provide opportunity for great ingenuity in the utilization of known properties of the glass-forming oxides, but their incorporation in appropriate illuminating mechanisms is largely a matter of engineering skill rather than of fundamental glass development.

More fundamental is the development of glasses with new properties of selective absorption and transmission which permit admission into the room of visible sunlight with or without the ultraviolet (short) or infrared (long) wave lengths or these may be admitted alone, for hospital, photographic or other purposes. There are many such glasses on the market, some colored, some plain, but all with selective qualities which have been developed with great precision in this country in recent years. For example, Dr. W. W. Shaver[5] has lately published an account of certain glasses, recently developed, which when used as windowpanes, admit into the room less than half of the total solar radiation which falls upon them, while retaining practically full window visibility. This means that practically all of the infrared and perhaps some of the ultraviolet have been cut off and absorbed by the glass while the visible spectrum passes freely through. Something like the opposite effect is produced by other glasses which permit free transmission only to ultraviolet or infrared and are of immense value in various hospital treatments.

Such are the questions and relationships which are determined mainly by the glass composition, or, to put it a little differently, this is the way the problem of glass development appears today from the chemical side. But it happens that in very recent years we have heard rather more about the physical properties of glass—glass strong enough to support an elephant on a shop windowpane. Perhaps the first thought in this direction still derives from chemical antecedents, namely, to experiment with changes in the composition until a notably stronger glass is found, but all the extensive and varied experimentation through the years of the active half century just past has revealed no outstanding development in that direction. Changes in composition may alter the strength of the early glasses two or possibly threefold, but the resulting products do not promise to displace the metals for strength.

It was known to the French in the latter part of the last century that dipping almost any glass, while still hot, into an oil bath would temper it in such a way as to give to it either great weakness or great strength. If the tempering was extreme, the glass became so brittle as to fly into a fine powder if scratched. The Prince Rupert drop is a familiar laboratory example. A treatment something short of that, however, left the glass much stronger than before. And so, casehardening or tempering first came to be applied to glass fifty years or more ago, but very little use was made of it at that time. Today the taxicabs of Paris are required to use windshield glass (Securit) which is tempered so highly that if it is scratched or broken it will shatter into fragments three or four millimeters in size, largely cubical in shape, which cause no damage. In England a similar glass is used under the name of "Armourplate" and shows very great strength.

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[5] W. W. Shaver, "Windows—and Their Relation to Air-Conditioning Problems," *Refrigerating Engineering*, September, 1933, Vol. 26, p. 133.

In this country we have chosen a somewhat different method of approach to the windshield problem for automobiles by enclosing between two glass plates a plastic layer of high transparency. By this method of approach, if the windshield is broken, no fragments, either large or small, fly at all. And so, different groups have used different methods to reach this physical result of greater strength and less danger if or when breakage occurs.

I happened once to visit the great glass plant at Jena twenty-five years or more ago, when a large glass disk had just been taken out of an annealing furnace, after treatment to remove strain. When I reached the spot where it had stood, there was nothing left but radial fragments, pointed like sharp knives, scattered through a distance of 50 ft. in all directions from where the disk had stood. The strains had not been successfully removed.

This matter of the physical treatment of glass, of taking account of the strains that may be in it and their possible effects, both economically and socially, is one of vital importance. They may be removed by careful annealing after the now well-established theory of Adams and Williamson, or they may be controlled and utilized. One of the more recent developments of this latter sort, which serves to show how the strain factors have now been brought under control, is found in the so-called "top-of-stove ware" which has lately appeared in the market. This is a highly tempered glass in which the softening point is so high that the ordinary operations of cooking do not reach it; the initial stresses are temporarily relaxed when the dish is put on the fire, and so, the heating operation does not impose a danger. There are indispensable requirements associated with such treatment to insure success, namely, the glass composition must be so chosen that no ordinary heating will raise the ware to a temperature where the predetermined strain relations are permanently altered, and second these strain relations must be so carefully predetermined that nothing approaching a Prince Rupert drop results. Of course, if hot glass is plunged into oil, you understand that the outside surface "stays put" and the inside continues to contract, which produces tension within the glass, compression in the surface layer, and a considerable stress difference between body and surface. It is the absolute control of these stress relations both in magnitude and orientation which has made possible the remarkable demonstrations of glass strength to which reference has been made above. But first it is necessary to develop a glass of such composition that it will take and retain these accurately predetermined strain relationships, and this advance is itself a landmark in the progress of American glass development.

While we are considering strictly American developments, I am rather disposed to tell a War story—very briefly. About a month before we entered the great War in 1917, there was some degree of apprehension among members of the general staff of the army and others in authority in Washington lest we did not have immediately available all the resources necessary to an active participation therein, should such a course of action be determined upon; and one of the indispensable supplies in which a shortage was immediately discovered was optical glass. European nations had been in the trenches for nearly three years, the methods of warfare had altered rapidly in that time and in directions very different from those which had prevailed before. In the indirect fire of the new tactics very much more accurate and elaborate fire-control instruments were needed than had been usual in any experience of ours. It meant that we must have optical glass in several different varieties and in high perfection for use in gunsights, periscopes, range finders, officers' field glasses and the like.

As a matter of fact, we had already been making such optical equipment in this country, using imported glass, for the Canadian and British governments, for some time, but in 1917, when the matter came up for our own consideration, we had no imported glass left; it was even difficult to find samples of all of the varieties needed. In those somewhat trying days it suddenly occurred to a good many people that optical glass had never been made in this country in quantity. A few experiments had been made privately on a small scale, but there was no one available with skill and experience sufficient to begin the melting of glass on an adequate scale and in acceptable quality for optical purposes.

Optical glass differs greatly from ordinary glasses in its very high physical perfection, in its very high light transmission, in its accurate refractive index and other optical constants; in every detail it must be a precisely determined and perfect product. To add to our perplexity, in the European countries whence we had obtained the glasses hitherto used for such purposes, the processes of manufacture had been kept carefully secret and were nowhere on record. That was the situation which confronted us in 1917.

I cannot tell the whole story, even after this lapse of time, although it would be extremely interesting to do so. Suffice it to say that our silicate chemists, not having any of the mysterious information about the ingredients and the processes which had been kept secret, went to work exactly as only research men do; they analyzed the existing samples and assembled the indicated raw materials, they calculated the evaporation of alkali during the melting process and the kind and amount of material likely to be dissolved out of the containing vessel, and they estimated about what the initial composition would be which would yield the required product. Then they made up two other samples differing from the first by a few per cent in the most critical ingredient, melted the three samples under like conditions and plotted the curve representing their relationship one to another and to the prescribed sample. In almost every case the exact specifications for the glass desired fell within that row of three observations, and with it the cloak of mystery disappeared from the entire process. It was a simple research undertaking directed straight to its purpose by experienced research men who happened never to have seen a pot of optical glass before, and within two months after we actually entered the war, we were producing, at the rate of 4000 lb. per month, glass which was satisfactory to the established Government inspection service. Estimated requirements had been set at 2000 lb. per day, but this was merely a matter of men and plant capacity once the crucial problem of how to do it had been solved. In no single instance did we fail to provide the particular kinds of glass which were required for our war purposes.

This simply serves to illustrate what many of us already have learned from personal experience, namely, that secret processes are generally a cloak to cover ignorance rather than great wisdom. The War Department has since published its war experience in glass making and all the mystery is now gone out of it—presumably forever.

It remains for us to turn our attention to some of the technical processes in which America has shown such outstanding distinction in the glass industry. Continuous melting in tanks was begun originally in Germany late in the last century; but the continuous-melting tank has developed both fast and far in this country since the original discovery by Siemens. Automatic continuous feeding of the molten product out of the melting tank and into equally automatic forming machines which lay down finished bottles, fruit jars and miscellaneous containers of every shape and almost every size and color in an unbroken stream upon an endless belt, is almost exclusively an American development. Tubing and window glass also are drawn continuously from mandrels, forms or rolls with little need for finishing operations beyond annealing and cutting to size; even figured glass partitions are laid down by rolls in continuous process at many feet per minute. Likewise the incandescent light bulbs, now indispensable in every home, are made in this country entirely by automatic machinery developed here.

To the author the electric light bulb has always seemed a very wonderful object. Its extreme thinness to support atmospheric pressure of 15 lb. per sq. in., and its complete freedom from mold marks or flaws of any kind, commend it as one of the most refined products of the glassmaker's art.

In 1876, when Edison prepared to exhibit his first bulbs at the Centennial Exposition in Philadelphia, the glass portion seemed to offer the greatest difficulty of attainment and long and ingenious experimentation proved necessary to reach a result. The manner of it was this. The glassmaker reached in with his long iron tube or "blow-iron" and gathered from the surface of the molten glass a small quantity sufficient to form the bulb and something more. After manipulating the glass on the end of the iron to a symmetrical shape, he blew into it a small puff of air and swung the iron above his head to cause the glass to elongate. The mystery of freedom from surface unevenness or markings was solved by the peculiar type of mold used. The mold might be made of iron or other heat-resisting material, but it must be lined with a thin layer of fine-grained, porous charcoal, thoroughly wetted by dipping in water. Into this form set on the floor with its wet lining, the glass-blower lowered his elongated glass bubble. Of course the water carried by the wet charcoal became steam at once in the presence of the white-hot glass and against this even coating of steam the bulb was blown with firm air pressure, the tube being turned between the hands of the operator during the whole operation to insure that the hot glass should encounter no resistance other than the even coating of the steam cushion which then lined the mold. Thus it was that the bulbs carried no marks as bottles do, and if the glass was of suitable quality an even, thin wall was assured. For thirty years following the Centennial Exposition and Mr. Edison's first successful electric lamps this was the only method known through which satisfactory bulbs could be obtained. To imitate this operation by machine combinations seemed inherently impossible, so much of the personal skill and judgment of the operator in timing the several operations seemed to be necessary to insure a successful product.

The story of the eventual development of machines is too long and contains too many steps to be presented here. The first efforts were, naturally enough, to provide glass of strictly uniform temperature, since only thus could accurate timing and a uniform mechanical process be applied to it with success. The next effort was to imitate the successive operations of the hand-worker, including elongation by swinging in the open, a very precarious and often unsuccessful process when applied to fluid glass. Success was finally attained by a process not usual in the development of automatic machinery, namely, by completely abandoning all of these details developed out of the experience of the hand-worker and basing all operations upon the measured mechanical properties of molten glass at the various stages and temperatures through which it must pass. Briefly described the machine in use today is a "theoretical" development and not an embodiment of the hand-worker's skill. It receives a uniform stream of glass about 1 in. in diameter flowing vertically from an opening in a continuous-melting tank. This stream is received between two water-cooled metal rolls, one smooth like a pulley and the other with cup-like depressions at regular intervals upon its periphery. The cylindrical stream is thus converted into a flat ribbon, with biscuits of molten glass upon it at appropriate intervals, which is received upon a moving endless belt, each biscuit accurately placed over an opening an inch or more in diameter in the belt itself. The molten biscuits sag through these openings forming hollow pear-shaped drops, in general form not different from the final bulb. As the soft bulbs gradually increase in length in the progress of the belt, molds, with the usual wet charcoal lining and moving at the same speed, close about them from below, while from above tiny pumps also moving and capable of injecting puffs of air, close tightly down upon them. The molds below turn rapidly about the bulb to insure a uniform steam cushion and the air puffs increase in strength above. Thus the belt carries its ribbon of glass forward continuously with its biscuits still attached, each of which has now become a fully formed bulb within its mold. The motion is entirely continuous, without jar or interruption, and all operations are carried on as the bulbs move smoothly on their way. At the right moment the molds divide in turn and fall away leaving the finished bulbs still suspended from their ribbon, down through the holes in the belt. A moment later a small hammer strikes a blow near the point of contact between the suspended bulb and the steel belt where the chilled contact zone makes the break-off easy. After passing through the annealing fires the bulbs are let fall upon an endless belt of asbestos which carries them to the inspectors. A single machine now completely automatic delivers these bulbs at the rate of half a million in a 24-hr. day, where formerly a hand-worker with his helper produced perhaps 1200 in an 8-hr. shift.

These machines are strictly an American product, developed in the American labor-saving spirit and necessity. Some European countries and Japan, where labor is cheap and standards lower, still prefer the old hand methods.

Since the borosilicate glasses became available with their low coefficient of expansion, high softening temperature, and almost complete insolubility even in strongly acid solutions, the engineering applications of glass have expanded greatly. When to these conspicuous advantages there is added the possibility of well-controlled tempering by physical processes, we have a material, available for a vast number of manufacturing and engineering uses, of rugged dependability.

Certain of these new uses may be passed in brief review: One of the most interesting is the introduction of great pipe-line systems for the transportation of solutions, hot or cold, or petroleum products, under conditions such that the material which is being transported is at all times under direct observation, as is also the condition of the pipe line itself and of sediments which may be deposited along the way. It has even been considered practicable to lay such pipe lines by the mile overland for the mass transportation of liquid products. Their rugged resistance to corrosion, both from without and from within, in addition to the visibility, give to such pipe lines obvious advantages to be obtained in no other way.

In the same sense and for the capitalization of the same advantages, great towers have been erected for distillation, filtration, or absorption processes in which continuous observation of the progress of reaction is desirable and not otherwise available. It is also obvious that all such pipe lines and towers of glass can be more satisfactorily cleaned than metal lines used for similar purposes.

These borosilicate glasses have also been widely used for insulators, both pole and suspension, in place of porcelain, where a uniform quality of insulation and freedom from disturbances due to moisture are desirable. Not only does the glass surface offer the usual qualities of resistance to weathering, but their transparency to solar radiation provides unexpected advantages in avoiding excessive temperatures and some other minor exigencies of line-insulation service. Such insulation is said to be exceptionally desirable for line transmission in connection with radio broadcasting.

It would seem to be a somewhat daring proposal to attempt to substitute glass for building blocks in structures subjected to the hard usage of manufacturing plants. Nevertheless, this also has been successfully accomplished in recent years without sacrifice of essential qualities of strength and ruggedness and with some obvious and hitherto unavailable advantages. For example, hollow building blocks are now in daily use in which the air may be evacuated from the hollow spaces and, so, the conductivity of the blocks for heat greatly reduced. Of course this is true alike of summer heat from without and winter heat from within. Such glass insulating blocks serve admirably in side walls where light is to be admitted and heat transmission limited. Also, the artist has not failed to take advantage of this situation to beautify side walls with artistic designs in glass, where the major purpose is otherwise prosaic.

There is one development in the recent engineering uses of glass which is in no sense new but which has only recently found extended interest in use. I have in mind the glass fibers now widely used in loose thick mats for insulating walls and roofs of buildings against heat transmission. Although glass fibers have been made and used for decades for isolated purposes, usually in connection with laboratory experiments, no serious interest appears to have been taken in these until the last decade, although the advantages of an inorganic heat insulator which is not subject to the usual deterioration of organic materials is sufficiently obvious. Perhaps one reason for this may be recognized in the fact that the fibers of earlier days were relatively coarse compared with those now on the market. Also, the earlier processes were relatively expensive and somewhat too experimental for quantity production. Now these fibers are made of a fineness such that a pound of glass in fiber form would extend around the earth. Perhaps it has also escaped attention hitherto that the surface tension factor in these fine fibers increases the tensile strength of glass beyond a million pounds per square inch.

The primary uses of these fibers is in loose mats, several inches in thickness, which may be inserted in hollow walls or between the beams of roof trusses, indeed almost anywhere where space is available for its introduction. Even in old wooden houses glass wool may be introduced into the hollow walls without other preparation than boring a few holes and introducing the wool under the power of a blast of air.

Thus far the composition of the wool appears to be less important than its physical consistency. It may be made from the glass obtained by melting down certain rocks and blowing out fibers from the molten mass. Glass residues of varied origin and composition have also been successfully reduced to fiber and used in these situations. When American enterprise goes farther and insists on producing fibers so fine that they may be woven into cloth and handled freely without abrasive effects upon the hands or other body contacts, and in particular when colored glasses are so used, then attention requires to be given to the composition as well as to the mechanical properties of the product. Woven glass hats, I am told, are already on the market in attractive models acceptable to the refinement of feminine taste. Glass clothing has intriguing possibilities, not only because of its inorganic make-up, but because of its physical properties in such use.

Of course, this is not the first time in which such a thought has found a place in the human mentality. It is within the memory of some of the older of us that a Spanish princess of royal blood once visited this country upon an important occasion and included in her wardrobe a most attractive gown of woven yellow glass. This was more than forty years ago.

Latest in the domain of effective uses of borosilicates of the Pyrex type, and still foremost in public interest, is the great 200-in. telescope disk lately made for the California Institute of Technology for use on Palomar Mountain in southern California. The disk is 17 ft. in diameter, 26 in. thick, and is probably the largest single piece of glass ever successfully made for any purpose. It weighs about 20 tons. It is made in the form of a grid, like a waffle iron, except that the units of the grid structure are triangular and not rectangular. The purpose is to maintain rigidity and reduce weight, because in dealing with a mass of a magnitude never before attained, many of us were apprehensive that the optical surface imposed upon this glass for astronomical purposes, which must be correct within one-twentieth of a wave length of light, might easily become distorted by a slight sagging under its own weight as its position changed for observations in different parts of the sky. And so it was thought to retain the rigidity and decrease the weight by about one-half through these open grids at the back of the mirror.

There was another purpose, perhaps equally important, to be served by this open structure. If this disk had been solid, it would have been necessary to support it around the rim by a strap and from the back by adhering contact plates; with this open grid structure it was possible to provide multiple supports *in the plane of the center of gravity of the disk.* One hundred and forty-two of these are provided for at the intersecting angles of the triangular grid structure, each of which will have its own compensating weights such that the amount of strain on the glass will remain the same regardless of the angle to which the disk may be turned in observing the stars. With this ingenious mounting the strain upon the glass will nowhere exceed about 35 lb. per sq. in.

This disk is the greatest single undertaking, so far as I know, that has been attempted in the glass world. A special train was used to transport it to its destination, for the rather critical reason that the net clearance between the disk and the bridges across the country under which it must pass amounted to no more than 5 in.

After this achievement no dream seems too fantastic for eventual embodiment in glass: glass houses (for those who do *not* throw stones), glass boats, and even the glass coffins have already "come true."

FAILURE OF HEAT-TREATED STEEL WIRE IN CABLES OF THE MT. HOPE, R. I., SUSPENSION BRIDGE[1]

By W. H. Swanger[2] and G. F. Wohlgemuth[2]

Synopsis

In 1929, several months after the completion of the spinning of the cables, but before the completion of the bridge structure, fractures were discovered in the cable wires at the anchorages of the suspension bridge across Mt. Hope Bay between Bristol and Portsmouth, R. I., and it became necessary to dismantle the partially completed bridge to prevent its collapse. The National Bureau of Standards was invited, by those concerned with the construction of the bridge, to make such studies of the failed wire as would lead to an explanation of the cause or causes of failure. This paper is a report on the results of these studies.

The failed wire was a basic open-hearth steel, brought to a tensile strength of approximately 220,000 lb. per sq. in. by heat treatment, subsequently galvanized by the hot-dip process. The failed wire was replaced by a hot-galvanized, cold-drawn steel wire of approximately the same tensile strength. It was proposed that not only should the studies cover the conditions that caused the heat-treated wire to fail, but also an investigation of behavior of the cold-drawn bridge wire under the same conditions.

Extended studies of the microstructure of the failed wire did not disclose any evidence of defective material or faulty heat treatment to which the failure could be ascribed. Intensive search failed to disclose internal cracks or fissures as a possible cause of the failure.

The failures on the bridge had occurred at the anchorages, although the wire there was not different from that located elsewhere in the cables. It was evident, therefore, that the cause of the failure was associated with stress conditions at the anchorages. Yet the cold-drawn replacement wire subjected to the same load has not failed. Either the stress conditions in the heat-treated wire at the time of failure were different from those imposed upon the cold-drawn wire or the latter was better able to resist the stresses which caused failure of the heat-treated wire. The results of the investigation show that both conditions obtained.

The fractured surfaces of wires which broke on the bridge showed definitely, and almost without exception, the characteristics of fatigue fractures. This fact indicated that fluctuating stresses were the damaging stresses when the wires failed.

Fatigue tests indicated that the fatigue limits of both types of wire were approximately the same, and that the failure of the heat-treated wire was not caused solely by its low fatigue limit in comparison with its tensile strength.

Because of the high elastic limit of the heat-treated wire, provision had been made for "preforming" each wire, where it passed around the anchorage, to a loop of the same curvature as the anchor shoe so that each wire would then "lay dead" against the anchor shoe, with no residual elastic bending stresses in the wire. It was found that

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

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most of the loops around the anchor shoes did *not* have the curvature of the shoes. In fact, many of them had reversed curves and when constrained to the shape of the shoe would be subjected to higher elastic bending stresses than if they had not been preformed at all.

Numerous specimens of both types of wire—heat-treated and cold-drawn—were subjected to various ranges of fluctuating tensile load while suspended over circular supports of the same radius of curvature as the bridge anchorages. For all loading conditions the endurance of the cold-drawn wire was greater than that of the heat-treated wire. The difference was greatest at low loads. It is believed that the fluctuating tensile load tests described in the paper reproduced the mechanism of fracture of the wires on the bridge.

The way in which the failed wires were placed on the anchor shoes, the high elastic limit of the material, and the fluctuating loads on the wires while the cables were spun and the bridge structure was being erected combined to produce a *range* of elastic bending stresses in the wires at the anchor shoes. The uniformly fine-grained steel of the wire, surrounded by the brittle, low-strength zinc-iron alloy of the galvanized coating, and with numerous surface imperfections acting as points of stress concentration offered inadequate resistance to the repeated elastic bending stresses. Fatigue fractures originating at the surface of the wires were the result. There was definite evidence that the fibrous structure of the cold-drawn wire tended to turn aside such cracks, whereas the hard fine-grained structure of the heat-treated wire could not act in this way.

INTRODUCTION

In February, 1929, several months after the completion of the spinning of the cables but before completion of the bridge structure, fractures were discovered in the cable wires at the anchorages of the suspension bridge across Mt. Hope Bay, between Bristol and Portsmouth, R. I. During the next six weeks the number of broken wires increased to such an extent that it was necessary to dismantle at once the partially completed bridge to prevent its collapse. The wire on this bridge was radically different from the wire previously used for cables in suspension bridges. The outstanding difference was that its high strength (ultimate tensile strength 220,000 lb. per sq. in.) was obtained by quenching and tempering a carbon steel wire instead of by cold drawing the wire through a die. The wire was galvanized by the hot-dip process after heat treatment.

At about the same time that the Mt. Hope bridge was being erected, a somewhat larger suspension bridge, the Ambassador bridge, was being erected across the Detroit River, between Detroit, Mich., and Windsor, Ont., in which heat-treated wire was also used, processed in the same plant and at the same time as the wire for the Mt. Hope bridge. Shipments to both bridge sites were taken at random from the same warehouse. Shortly after the first failures were reported at the Mt. Hope bridge, similar failures were reported at the Ambassador bridge and it too was dismantled. The wire removed from both bridges was replaced with the conventional cold-drawn wire, and the bridges were completed shortly thereafter. No failures of wires in either bridge have been reported since their completion.

The failure of the wire in these two bridges naturally aroused a widespread public interest. No such failure had been encountered before in suspension bridge construction. The fact that the failed wire had been heat treated naturally focussed attention upon this departure from previous practice in the manufacture of suspension bridge cable wire as the source of the difficulty. Such a conclusion, if well founded, would cast doubt upon the reliability of all heat-treated steels, a corollary not in accord with observed fact.

It was agreed by all those involved in the construction of the two bridges that a detailed study of the heat-treated wire should be made to learn the facts that caused the failure. As an amicable settlement of the expense of changing over from the heat-treated to cold-drawn wire cables had already been made, a question of blame was not involved. The National Bureau of Standards was invited to undertake the investigation. Feeling that the situation was a matter involving safety of the public, the Bureau agreed to undertake the investigation and to publish the results.

An ample quantity of the heat-treated wire was obtained under the supervision of representatives of the Bureau at the time the cables of the Mt. Hope bridge were dismantled. Included in this were all of the wires around each of the anchorages at both ends of the bridge. A supply of the cold-drawn replacement wire was also secured, together with coils of the rod and wire at the various stages of the manufacturing process for both types of wire.

The span of the Ambassador bridge was somewhat longer, and the number of wires per cable was somewhat greater than in the Mt. Hope bridge but the wire for one bridge was indistinguishable from that used on the other, and wire removed from the Ambassador bridge was not used in the investigation. This discussion is therefore confined to the conditions at the Mt. Hope bridge but is believed to apply equally to both bridges.

The problem was two-fold. It was felt that not only should studies be made to learn the conditions that caused the heat-treated wire to fail, but that the behavior of the cold-drawn wire under the same conditions should also be investigated.

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H. L. Whittemore, L. B. Tuckerman, W. R. Osgood, and C. S. Aitchison of the Engineering Mechanics Section, and H. W. Gillett, J. R. Freeman, Jr., and R. L. Dowdell, formerly of the Division of Metallurgy of the National

Bureau of Standards, made the preliminary inspections at the bridge site, supervised the selection of the materials to be investigated, and initiated the investigation at the National Bureau of Standards. Useful advice and assistance in the performance of the various mechanical tests were furnished by the Engineering Mechanics Section.

C. A. McCune, formerly Director of Research of the American Chain Co., and the Page Steel and Wire Co., furnished details of the manufacturing process and results of a number of studies of the properties of the heat-treated wire, as well as additional material for the investigation.

E. E. Thum, Editor, *Metal Progress*, and F. E. Schmitt, Editor, *Engineering News-Record*, as representatives of the engineering profession, were active in urging that a thorough and impartial investigation of the facts causing the failure be made. Mr. Thum maintained a keen interest throughout the course of the investigation and many helpful suggestions were received from him.

TABLE I.—LENGTH OF MAIN SPAN AND SPECIFIED TENSILE STRENGTH OF CABLE WIRES.

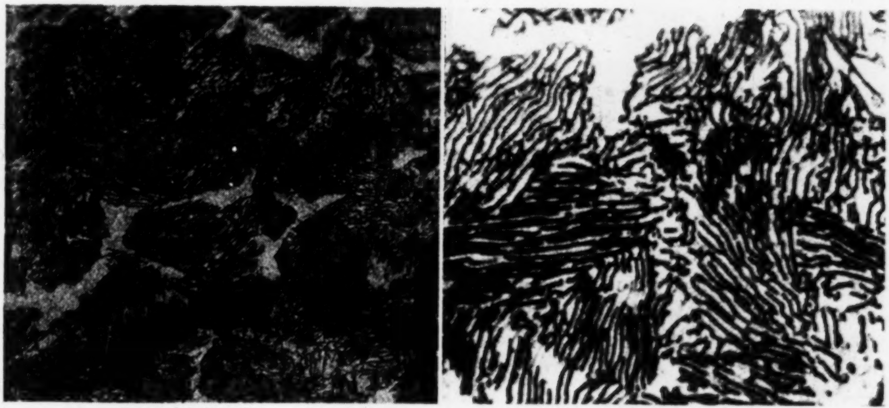
Name of Bridge	Date of Erection	Length of Main Span, ft.	Tensile Strength, lb. per sq. in.
Brooklyn	1870–1883	1596	160 000
Williamsburg	1896–1903	1600	200 000
Manhattan	1901–1910	1470	210 000
Bear Mountain	1923–1924	1632	215 000
Delaware River, Philadelphia to Camden	1922–1926	1750	215 000
George Washington	1927–1932	3500	225 000
San Francisco-Oakland Bay	1933–1937	2310 (2 spans)	220 000

To all of the above the authors of this report make grateful acknowledgment.

MANUFACTURE OF THE HEAT-TREATED WIRE

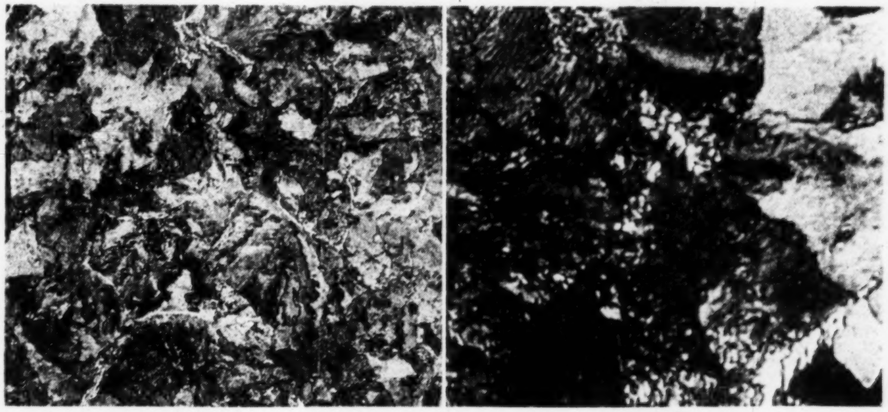
Cold-drawn steel or iron wire has been used for the cables of all large suspension bridges except the original cables of the Mt. Hope and Ambassador bridges. The specified tensile strengths of the cable wires in some of the important suspension bridges built since 1880 are listed in Table I.

The increased tensile strengths of the wire, shown in Table I, were largely the result of increase in amount of plastic deformation in drawing. This was made possible by a special type of heat treatment, peculiar to drawing high-strength steel wire, and called, in that industry, "patenting." Hot-rolled rod of the type of steel used for bridge wire normally has a two-phase structure known as pearlite. In this condition the steel cannot withstand the severe plastic deformation necessary to produce the desired strength. In the "patenting" heat treatment the rod is heated to a temperature at which the two-phase structure disappears, then cooled rapidly



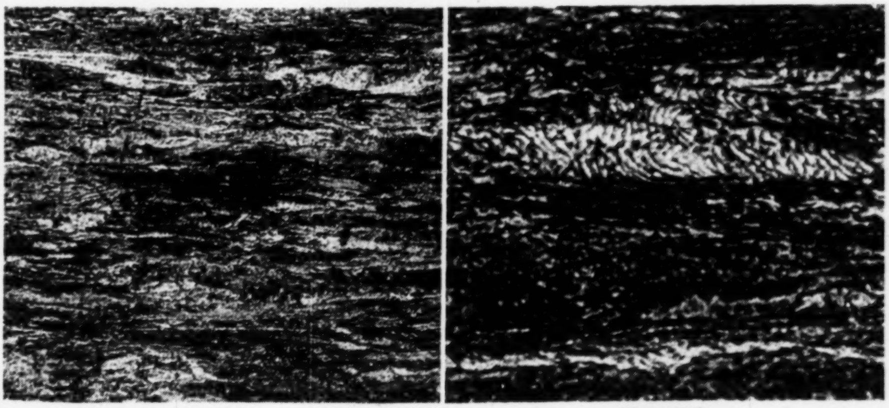
(*a*) × 500. (*b*) × 2000.

FIG. 1.—Structure of Hot-Rolled Rod for Cold-Drawn Bridge Wire, Pearlitic.
Etched, saturated solution, picric acid in 95 per cent alcohol.



(*a*) × 500. (*b*) × 2000.

FIG. 2.—Structure of "Patented" Rod for Cold-Drawn Bridge Wire.
Etched, saturated solution, picric acid in 95 per cent alcohol.



(*a*) × 500. (*b*) × 2000.

FIG. 3.—Structure of Wire Drawn 5 Holes from Patented Rod.
Etched, saturated solution, picric acid in 95 per cent alcohol.

to a temperature in the range approximately 900 to 1100 F. (480 to 595 C.). The structure produced at this temperature is retained when the rod is cooled to room temperature and may be considered as having properties of a single-phase structure. The process of producing it has been called "sorbitizing." In this condition the steel withstands the severe drawing deformations that produce the high strength without a deleterious decrease in ductility.

A second, but nevertheless important, function of the patenting treatment is to eliminate the non-uniformity in structure of the hot-rolled rod arising from ingot segregation, variations in rolling temperature, and differences in rate of cooling after rolling. The uniformity in strength and other properties of cold-drawn bridge wire depend to a most important degree upon the skill with which the patenting treatment of the rod has been controlled.

TABLE II.—TENSILE PROPERTIES AND HARDNESS OF THE HEAT-TREATED WIRE THAT FAILED AND THE COLD-DRAWN WIRE WHICH REPLACED IT.

Material	Tensile Strength, lb. per sq. in.	Rockwell Hardness, "C" Scale	Elongation, per cent	Reduction of Area, per cent
HEAT-TREATED WIRE				
Hot-rolled $\frac{3}{8}$-in. rod	114 000	17	10	26
1 hole drawn wire, bright, 0.193-in. diameter	153 000	27	2	20
Heat-treated and galvanized wire	223 000	46	6	38
COLD-DRAWN WIRE				
Hot-rolled $\frac{3}{8}$-in. rod	120 000	15	11	25
Patented $\frac{3}{8}$-in. rod	170 000	32	7	36
5 hole drawn wire, bright, 0.193-in. diameter	247 000	40	3	41
Galvanized wire	223 000	41	6	40

The cold-drawn wire used to replace the heat-treated wire in the Mt. Hope and Ambassador bridges was drawn through 5 dies from a hot-rolled and patented rod, $\frac{3}{8}$ in. in diameter, to a final diameter of 0.192 in., without intermittent annealing.

The pearlitic structure of the hot-rolled rod is shown in Fig. 1 and the structure produced by the patenting treatment, in Fig. 2. The structure of the drawn wire before galvanizing is shown in Fig. 3. The structure of cold-drawn bridge wire is one produced by heat treatment, but altered in configuration, by cold working. The galvanizing process did not materially change the structure of the drawn wire, although its tensile strength and hardness were reduced, as is shown in Table II, where the tensile strength, hardness, elongation, and reduction of area are given for the wire in its successive stages from hot-rolled rod to galvanized wire.

The method of manufacture of the heat-treated wire differed widely from that just outlined for cold-drawn bridge wire. The process was originated by the American Chain Co. and the wire was made in the plant of the Page Steel and Wire Co., at Monessen, Pa. Full details of the process were furnished by this company to the National Bureau of Standards, but the production unit was not in operation at any time during the Bureau's investigation.

The steel, made in basic open-hearth furnaces, was received at the wire mill in the form of billets 4 by 4 in. square in section by 72 in. long, weighing about 300 lb. each. The total number of billets processed into wire for both bridges was 29,224. Of these, 18,796 billets from 60 heats were manufactured by the Central Alloy Steel Co. of Massillon, Ohio, and 10,428 billets from 27 heats by the Cambria plant of the Bethlehem Steel Co. at Johnstown, Pa. The ingots from which the billets were rolled all had been cast in "big-end-up" molds. The billets received from the Central Alloy Steel Co. had been chipped, those from the Bethlehem Steel Co. had not, but obvious defects were chipped from these billets in the wire mill. Cross-sections from the ends of the billets were deep-etched and inspected to detect segregation and pipes in the billets.

TABLE III.—COMPOSITION OF THE STEEL OF THE HEAT-TREATED WIRE.

	CAMBRIA BILLETS	CENTRAL ALLOY BILLETS
Carbon, per cent	0.71 to 0.83	0.72 to 0.81
Manganese, per cent	0.45 to 0.61	0.37 to 0.56
Phosphorus, per cent	0.014 to 0.030	0.016 to 0.024
Sulfur, per cent	0.025 to 0.037	0.019 to 0.038
Silicon, per cent	0.14 to 0.27	0.10 to 0.20

Carbon, manganese, phosphorus, sulfur and silicon contents of the steel were determined by chemical analyses made in the wire mill laboratory on samples from 3 billets from each heat. The specification requirements for chemical composition of the steel were as follows:

Carbon	0.70 to 0.80 per cent	Silicon	0.12 to 0.20 per cent
Manganese	0.40 to 0.60 per cent	Chromium	0.10 per cent, max.
Phosphorus	0.04 per cent, max.	Nickel	0.10 per cent, max.
Sulfur	0.04 per cent, max.	Copper	0.10 per cent, max.

In Table III are shown the ranges in composition determined by analyses of samples from billets of each heat from both sources. Each value is an average of three determinations on one heat. The maximum spread in the three determinations of carbon content for any one heat was 0.12 per cent for the Cambria billets and 0.07 per cent for the Central Alloy billets. The maximum spread in manganese content was 0.03 per cent for the billets from both sources. It was considered that the uniformity in composition of the steel was satisfactory.

The billets were rolled on a continuous Morgan mill to No. 4 gage (0.225-in. diameter) rod in the plant of the Page Steel and Wire Co. The billets were heated to the desired rolling temperatures in a continuous, oil-fired furnace. As no intermediate reheating was done during rolling, the back ends of the rods generally were finished at a lower temperature than the front ends. At times it was noted that the finishing temperature was below the critical temperature, so that cold work hardening was induced in portions of some of the rods by the rolling operation.

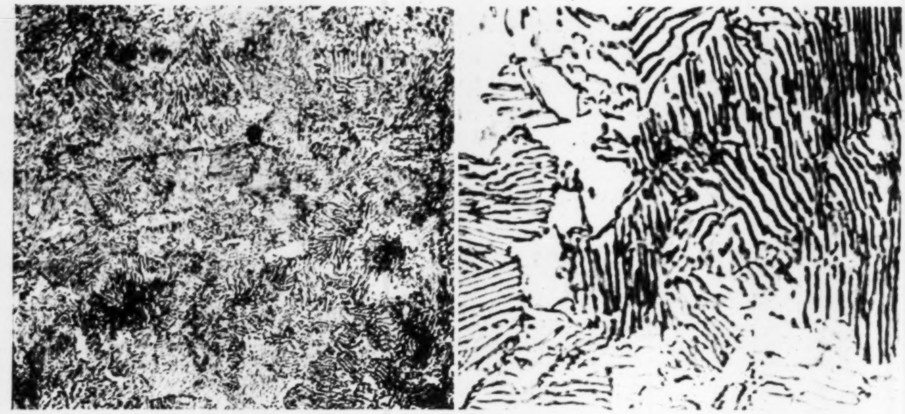

(*a*) × 500. (*b*) × 2000.

FIG. 4.—Structure of Hot-Rolled Rod for Heat-Treated Wire, Pearlitic.
Etched, saturated solution, picric acid in 95 per cent alcohol.

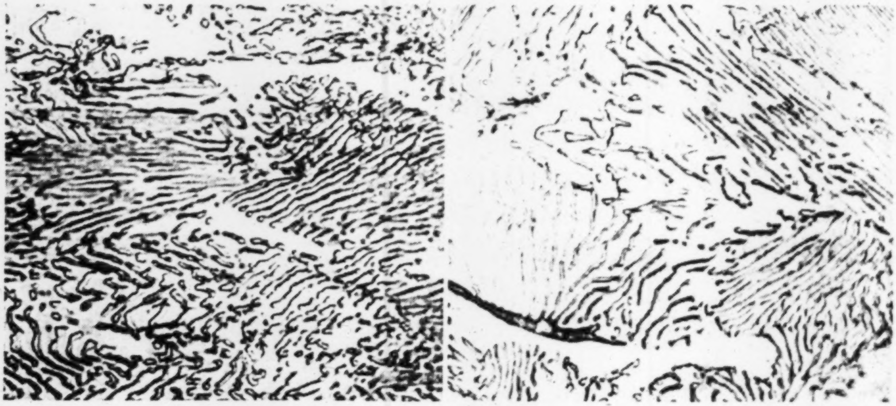

(*a*) × 500. (*b*) × 2000.

FIG. 5.—Structure of Wire Drawn 1 hole from Hot-Rolled Rod.
Etched, saturated solution, picric acid in 95 per cent alcohol.

The rods were coiled from the rolls and then pickled. The pickling solution was maintained at a 5 per cent sulfuric acid content (by weight) and at a temperature of 135 F. (55 C.). A thiourea inhibitor was added to the pickling bath. After pickling, the coiled rods were washed with water, then dipped into a lime vat and finally baked in coal-fired furnaces for 8 to

12 hr. at 300 F. (150 C.). Some of the coils, before being coated with lime, were dipped into a dilute copper sulfate solution where they acquired a light copper coating. Others were sprayed with water until they acquired a thin but tightly adhering rust coat. Both of these coatings together with the lime coat acted as lubricants in the drawing operation. Grease or soap was used as additional lubricant. The lime coat also neutralized any pickling acid not removed by washing. The hot-rolled rod was drawn through tool steel dies to No. 6 gage (0.192-in. diameter) wire, some of it in one draft, some in two drafts. The reduction of area from rod to wire was 27 per cent.

TABLE IV.—SEQUENCE OF OPERATIONS IN HEAT TREATMENT OF WIRE.

Unit	Temperature, deg. Fahr.	Temperature, deg. Cent.	Time at Temperature, sec.	Operation	Interval to Succeeding Operation, sec.
No. 1 Lead bath	1325 to 1350	720 to 730	24	preheat	2 to 4
No. 2 Lead bath	1550	845	35	heating	2 to 4
No. 3 Lead bath	1250	675	33	hot-quench	2 to 4
No. 4 Lead bath	1550	845	37	heating for quenching	5
No. 5 Oil bath	225 to 275	105 to 135	90	quenching	10
No. 6 Lead bath	810 to 850	430 to 455	18	tempering	..
No. 7 Acid bath	150	65	..	cleaning	75
No. 8 Wash water					
No. 9 Flux					
No. 10 Zinc bath	850	455	35	galvanizing reeling	..

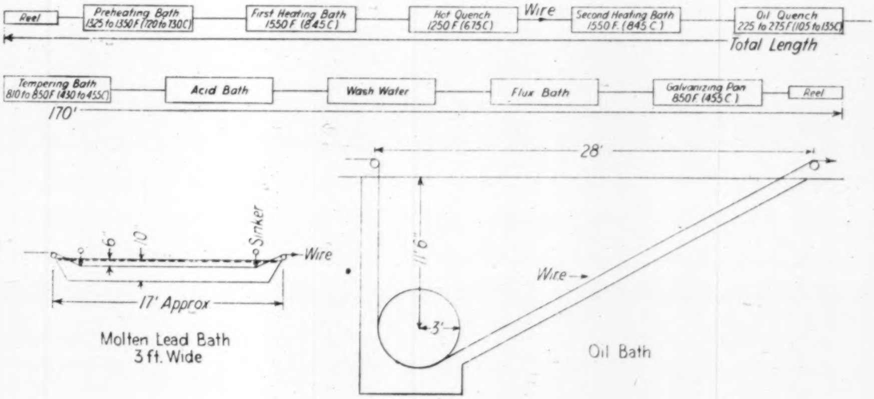

Reel
Preheating Bath 1325 to 1350 F (720 to 730 C)
First Heating Bath 1550 F (845 C)
Hot Quench 1250 F (675 C)
Wire
Second Heating Bath 1550 F. (845 C)
Oil Quench 225 to 275 F (105 to 135 C)
Total Length
Tempering Bath 810 to 850 F (430 to 455 C)
Acid Bath
Wash Water
Flux Bath
Galvanizing Pan 850 F (455 C)
Reel
170'
28'
9"
10"
Sinker
Wire
11'6"
Wire
3'
17' Approx
Molten Lead Bath
3 ft. Wide
Oil Bath

FIG. 6.—Flow Sheet of Heat Treating and Galvanizing Unit.

This drawn wire was quite different from the conventional cold-drawn bridge wire, both in physical properties and metallographic structure. The metallographic structure of the wire, as drawn, was not greatly different from that of the hot-rolled rod, as is shown in Figs. 4 and 5. In both rod and wire the structure is lamellar pearlite, not clearly resolved at 500 diameters magnification. In the drawn wire the inclusions and lamellae are oriented and somewhat elongated in the direction of drawing.

The unique feature in the manufacturing process was the next procedure in which the drawn wire was heated to a temperature above the critical temperature, quenched in oil, tempered to the desired combination of strength and ductility, cleaned, and galvanized in one continuous, uninterrupted passage.

The sequence of operations is summarized in Table IV, and shown graphically in Fig. 6. The wire was heated in a series of molten-lead baths to the quenching temperature, 1550 F. (845 C.). From this temperature it was cooled in an oil bath to a temperature in the range 225 to 275 F. (105 to 135 C.) for approximately 90 sec., and then, after a lapse of about 10 sec. during which it was exposed to the air, it was reheated rapidly in a lead bath to a temperature in the range 810 to 850 F. (430 to 455 C.) for approximately 18 sec. From this temperature the wire was cooled to about 150 F. (65 C.) for about 75 sec., during which it was cleaned, and then again reheated, in the galvanizing bath, to about 850 F. (455 C.) for about 35 sec. From this temperature the wire was cooled rapidly to room temperature and subsequently had no further heating by artificial means. The structure typical of all of the heat-treated wire examined microscopically is shown in Fig. 7.

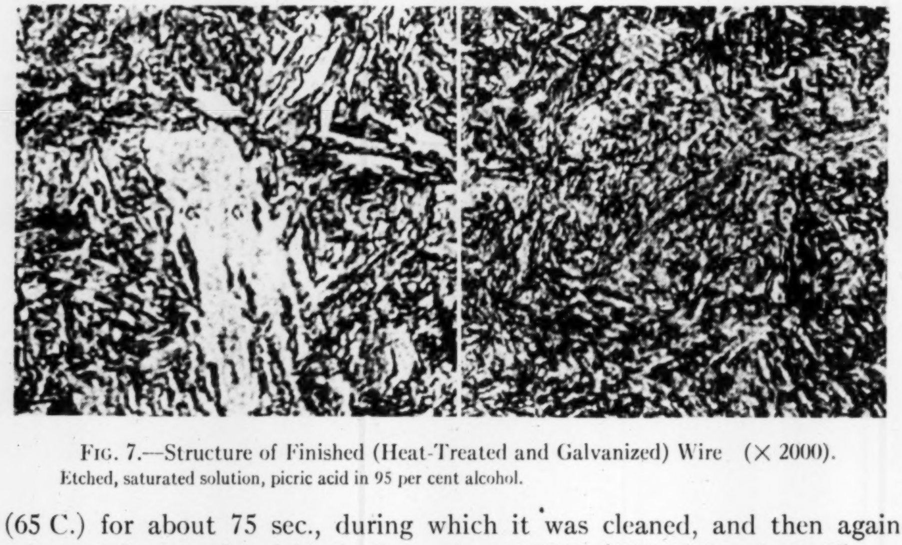


FIG. 7.—Structure of Finished (Heat-Treated and Galvanized) Wire (× 2000). Etched, saturated solution, picric acid in 95 per cent alcohol.

There is no direct evidence on the actual structure of the steel at any of the intermediate stages of the heat treatment. Discussions on the transformation of austenite by Lewis[3] and by Davenport and Bain[4] throw some light on the subject. Lewis worked with the steel used for the bridge wire, and steel B in the paper by Davenport and Bain was approximately the same. The results given in both papers and of studies made at the wire mill indicate that in the 90 sec. during which the wire was at the temperature of the oil-quenching bath, the austenitic phase was retained without formation of martensite, and that the structure produced in the tempering bath was obtained directly from austenite without intermediate formation of martensite. It is certain that if any martensite had been produced in the oil quench it would have been transformed in the tempering bath, and probably would be indistinguishable from the directly transformed austenite.

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[3] D. Lewis, "The Transformation of Austenite into Martensite in an 0.8 per cent Carbon Steel," *Journal*, Iron and Steel Inst. (British), Vol. CXIX, No. 1, p. 427 (1929).

[4] E. S. Davenport and E. C. Bain, "Transformation of Austenite at Constant Subcritical Temperatures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Iron and Steel Division, Vol. 90, p. 117 (1930).

During the 10-sec. interval between the oil-quenching bath and the tempering bath, when the wire was exposed to the air, it is possible that some martensite was formed. If so, it was transformed in the tempering bath.

However, if an appreciable amount of martensite had been formed in the quenching bath the wire would have been brittle and trouble would be expected in carrying the wire around the drum in the oil bath and over the roller at the exit end of the oil bath (Fig. 6). Such trouble was not experienced during the normal operation of the heat-treating unit. On occasions when, because of breakdown, the wire was halted in the unit for periods of 5 min. or more, it was found that the wires around the drum and over the roller at the exit end had taken a permanent set, were very hard and brittle, and would snap off without pulling straight. If any martensite was formed during the passage of the wire over the roller between the oil tank and tempering pan it was not sufficient to cause breaks in the wire.

If any martensite was formed during the time the wire was in the cleaning unit, at 150 F. (65 C.) for 75 sec., it likewise was not sufficient to cause breaks in the wire, and was tempered in the galvanizing bath. The additional tempering the wire received in the zinc bath accomplished a slight softening, probably as the result of an agglomerating effect on the fine constituent of the directly transformed austenite.

The question as to whether or not the structure in the steel when the wire was taken from the heat-treating unit was retained permanently is probably of more significance than is the structure at any stage during the heat treatment. This point was investigated by thermomagnetic analysis methods as described in a series of papers by Sanford and Ellinger of the National Bureau of Standards.[5, 6, 7, 8, 9] The first of these papers describes

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[5] R. L. Sanford, "Apparatus for Thermomagnetic Analysis," National Bureau of Standards *Journal of Research*, Vol. 2, p. 659 (1929).

[6] R. L. Sanford and G. A. Ellinger, "Thermomagnetic Analysis and the A_0 Transformation in 0.75-per-cent Carbon Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 83 (1931).

[7] R. L. Sanford and G. A. Ellinger, "Thermomagnetic Phenomena in Steel and Their Application in the Study of Tempering of Quenched 0.75 per cent Carbon Steel," *Transactions*, Am. Soc. Steel Treating, Vol. 20, No. 3, September, 1932, p. 263.

[8] G. A. Ellinger, "Thermomagnetic Investigation of Quenched 0.75 per cent Carbon Steel," National Bureau of Standards *Journal of Research*, Vol. 7, p. 441 (1931).

[9] G. A. Ellinger and R. L. Sanford, "Prolonged Tempering at 100 C. and Aging at Room Temperature of 0.8 per cent Carbon Steel," National Bureau of Standards *Journal of Research*, Vol. 13, p. 259 (1934); also *Transactions*, Am. Soc. Metals, Vol. 23, No. 2, June 1935, p. 495.

the thermomagnetic method of analysis, peculiarly applicable to show the changes in constitutional structure of a quenched carbon steel while it is being tempered. The 0.75 or 0.80 per cent carbon steel used in the work reported in the other papers was the steel used for the heat-treated bridge wire. It can be concluded from the results of Sanford's and Ellinger's work with this steel that the wire after leaving the galvanizing bath, and having cooled to room temperature, was in a permanently stable state of a completely transformed austenite (or tempered martensite) that would undergo no further structural change unless it were again heated to at least the temperature of the galvanizing bath, 850 F. (455 C.).

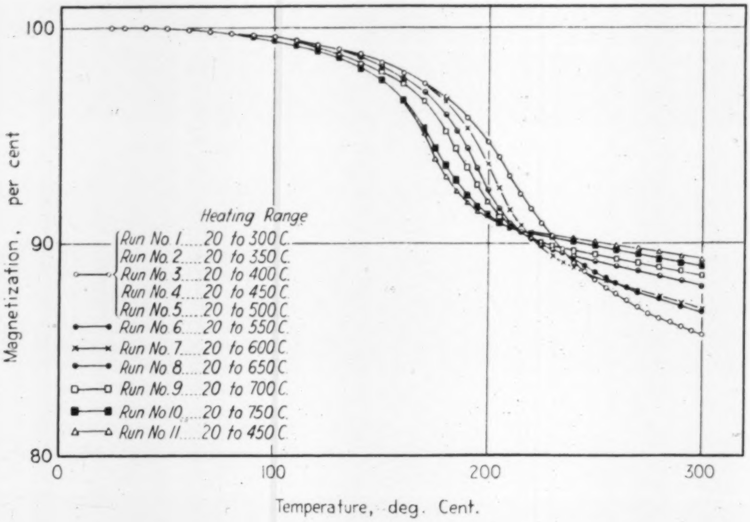

FIG. 8.—Change in Magnetization with Increase in Temperature for Specimen of Wire from Bridge, Heated as Shown.

This conclusion is corroborated by a set of previously unpublished thermomagnetic measurements by Ellinger, illustrated by the curves in Fig. 8. The material was a piece of wire from the Mt. Hope bridge adjacent to a fracture. The curves show the changes in magnetization as the specimen of wire was heated to various temperatures, held at temperature 15 min., and then cooled to room temperature. The changes that are significant of a change in constitutional structure occur in the range to 300 C. and for this reason the curves are given for this range only.

There was no difference in rate of change of magnetization between 20 and 300 C. for the first five runs in which the specimen had not been heated beyond 450 C., the temperature of the tempering baths. On run No. 5 the specimen was carried to 500 C. In run No. 6 there was a difference between 240 and 300 C. in the rate of change from the previous runs. In the succeeding runs, Nos. 7, 8, 9, 10 and 11, there was a progressively higher reading at 300 C. and the maximum slope of the curves, which may be considered as the A_0 transformation, was at progressively lower temperatures.

Another series of measurements by Ellinger was made on a set of 4 specimens from a coil of the drawn wire that had not been heat treated. The specimens were heat treated in the laboratory according to the times and temperatures shown in Table IV. Measurements were made of the change in magnetization with increase in temperature to 300 C. on one specimen immediately after it was removed from the tempering bath. Similar measurements were made on the remaining specimens after they had aged at room temperature for 24 hr., 72 hr., and 1 week.

The close agreement in the changes in magnetization of these specimens, shown by the curves, Fig. 9, which are typical of the results of many additional experiments of the same type, show plainly that the wire was in the tempering baths a sufficient length of time to bring about all of the structural changes in the quenched steel that could occur at the tempering temperature, and furthermore, that no changes in the constitutional structure of the wire occurred spontaneously shortly after it left the heat-treating units. It is believed that the wire was in the same condition, structurally, when it failed on the bridges as when it left the mill, and is today in that same condition.

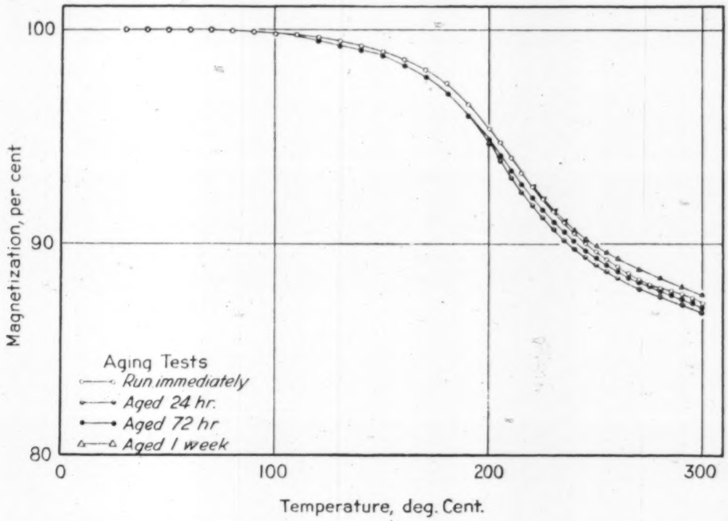

FIG. 9.—Change in Magnetization with Increase in Temperature for Specimens of Heat-Treated Wire (Heat Treated in Laboratory) Aged for Various Periods.

INSPECTION AND TESTING AT THE WIRE MILL

The coils of wire after removal from the 5-ft. winding reels at the end of the heat-treating and galvanizing unit were tied, separately, with soft iron wire and transported to the testing room where samples were cut from each end of each coil. The physical properties specified for the heat-treated wire were:

Tensile strength	220 000 lb. per sq. in.
Yield point (stress to produce 0.75 per cent elongation in 10 in.)	190 000 lb. per sq. in.
Elongation in 10 in	4 per cent
Reduction of area	30 per cent

It was also required that the wire withstand, without fracture, wrapping on a mandrel 4 times the wire diameter.

The coils which were accepted by the inspectors were sealed and then stored in a warehouse until reeled for transportation to the bridge sites. Of the 29,224 coils of wire completely processed, approximately 4000 were rejected in the testing room or on the processing unit. Rejection of about half of these coils was due to defects in the galvanizing. The remainder was rejected because of failure to meet the specified physical properties, or because they broke on the processing unit. The cause of failure to meet the specified physical properties was in most cases traced to surface defects in the rolled or drawn wire, such as laps, seams, overfills, and underfills that happened to be located in the test samples. In a small number of rejected coils the test samples were found to contain segregations. The cause of most of the failures on the processing unit was not ascertained in the wire mill. It was noted, however, that the fractures invariably originated at the surface and separation took place without distortion or reduction of area adjacent to the fractures.

Similar fractures were obtained on many coils shortly after they were removed from the heat-treating unit. Many such breaks were not discovered until the coils were taken from the warehouse to be wound on the 7-ft. reels for shipment to the bridge sites. This condition naturally caused concern about the soundness of the entire lot of wire. As a precautionary measure, each coil, before it was wound on the shipping reel, was passed through two sets of staggered rolls, somewhat like a wire-straightening machine. One set of 5 pairs of rolls bent the wire back and forth in a vertical plane, and the succeeding set of 5 pairs bent it in a horizontal plane. It was believed that the wire which did not break in passing through the staggered rolls was sound and acceptable for use on the bridges. Of the total number of 24,935 coils accepted by the inspectors and stored in the warehouse for periods of 2 weeks to 6 months, 337 were found with breaks. Breaks were produced in the staggered rolls in only 20 more. For various other reasons, 372 additional coils of the galvanized wire were not shipped, leaving a total number of 24,206 coils that were wound and shipped to the bridge sites.

The separate coils were spliced end to end with threaded sleeves as they were wound on the reels, each of which contained 10,000 lb. of wire, or about 30 coils.

Attempts were made in the wire mill to discover the conditions that caused the breaks in the wire during processing and shortly after the wire was placed in the warehouse. The identity of each coil had been maintained throughout the process, from billet to finished wire. The physical properties of the wire in the broken coils were apparently the same as those of unbroken coils. It was thought that reeling the wire from the galvanizing pan on 30-in. diameter coils imposed too severe a stress on the high yield strength wire, and 5-ft. diameter reels were substituted. However, similar breaks occurred on the larger coils. Breaks in the coil could not be correlated with deviations in chemical composition nor with imperfections in the billets. The percentage of warehouse breaks was twice as high in coils from the chipped billets as it was in coils from unchipped billets. No correlation was possible between prevalence of breaks in the coil and the sporadic variations in the operating conditions of the wire drawing, heat treating, acid cleaning or galvanizing subunits. It was noted that the number of breaks was greatest in the wire with the most pronounced surface defects. Of even more significance was a set of 74 coils run through the heat treating unit but not galvanized. Some of these coils were by-passed around the acid cleaning and fluxing tanks and passed through the galvanizing pan without taking on a zinc coating. Others were passed through the acid and subsequently baked, but were not passed through the galvanizing pan. Others were by-passed around both the acid tanks and galvanizing pan. No breaks occurred in any of these wires when reeled on 30-in. diameter coils.

In considering these results, hydrogen embrittlement is immediately thought of as a possible cause of the early breakages in the coils. The possibly damaging effect of the presence of the zinc coating in this connection cannot be considered very well by itself, because the wire could not be galvanized without first having been exposed to the acid cleaning treatment.

Hydrogen embrittlement in steel is known to be a transitory effect, disappearing in time, even at room temperatures. All of the wire that went into the Mt. Hope bridge was processed between February 9 and June 22, 1928. The first wire was wound on reels for shipment to the bridge on August 15 of that year. The minimum time in storage for any of this wire was therefore 53 days. Whether or not the wire recovered, in this time, from any damaging effect of hydrogen absorbed or adsorbed during the acid cleaning before it was galvanized cannot be determined now. If the wire was embrittled by hydrogen sufficiently to break when coiled on 30 or 60-in. diameter coils, or shortly thereafter, and if this embrittling effect had not disappeared when the wire was wound on the shipping reels, it is reasonable to expect that many breaks would have occurred in the staggered rolls, where it was bent back and forth on smaller radii of curvature than in the coils. Yet breaks were obtained in only 20 coils out of a total of over 24,000.

Numerous 30-in. diameter coils have been made from straight lengths of wire from the original cables of the Mt. Hope bridge. To date, not a single break has occurred in any of them. Anticipating somewhat the discussion in the following parts of this paper, this same wire, when subjected to stresses of the same sort imposed on it in the bridge cables, will fail with the same type of fractures that occurred on the bridge.

Although it is possible that hydrogen embrittlement may have been an important factor in causing the fractures in the coils in the wire mill, it is believed with confidence that the fractures on the Mt. Hope bridge are not to be ascribed to the same cause.

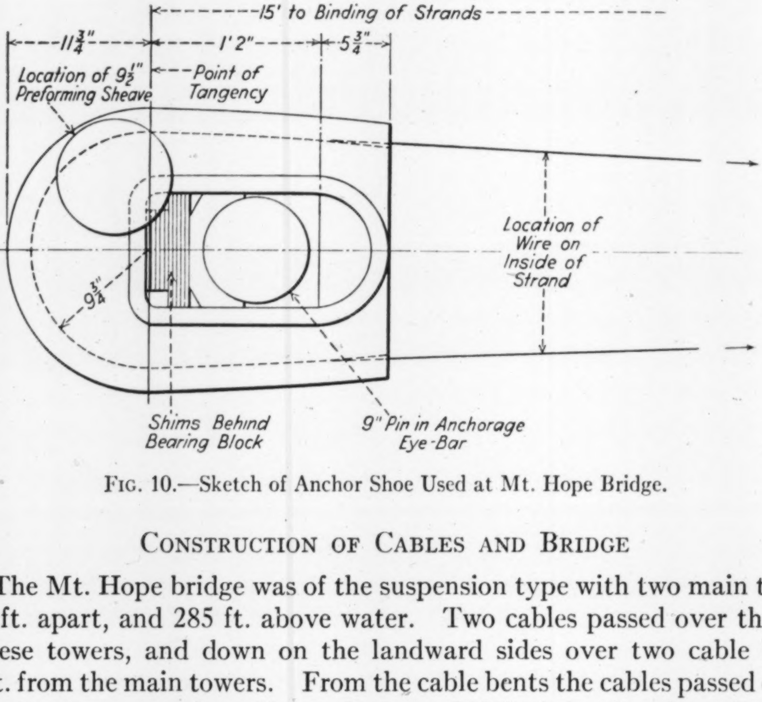
15' to Binding of Strands
11¾"
1' 2"
5¾"
Location of 9½" Preforming Sheave
Point of Tangency
Location of Wire on Inside of Strand
9¾"
Shims Behind Bearing Block
9" Pin in Anchorage Eye-Bar

FIG. 10.—Sketch of Anchor Shoe Used at Mt. Hope Bridge.

CONSTRUCTION OF CABLES AND BRIDGE

The Mt. Hope bridge was of the suspension type with two main towers 1200 ft. apart, and 285 ft. above water. Two cables passed over the tops of these towers, and down on the landward sides over two cable bents, 500 ft. from the main towers. From the cable bents the cables passed downward at a steeper angle to the anchorages, 220 ft. from the pivoted towers. The horizontal distance between anchorages was therefore approximately 2650 ft.

The bridge was designed so that there was no relative motion between the cables and the tops of the supporting towers. Changes in length of the cables, which had fixed ends, caused changes in their slopes on the spans. The cable bents, pivoted on their bases could readily accommodate themselves to such changes. The main towers were flexible and designed so that their tops could be deflected 11 in. on both sides of the center position, without permanent distortion of the towers.

Each cable consisted of 7 strands of parallel wires, compacted to a circular cross-section 11 in. in diameter. Each strand contained 350 wires, 0.195 in. in diameter. In the construction of these cables there were a number of departures from the conventional methods for spinning, described elsewhere.[10, 11] The cables rested on the tops of the supporting towers in large steel castings, or saddles.

At each end of the strand, the wires were looped around a steel casting or anchor shoe, shown in Fig. 10. The anchor shoe was joined, by a heavy pin, to an eye-bar which in turn was held by a "dead-man" embedded in the concrete anchor mass. Customarily the loops are carried around the anchor shoe in a horizontal plane, and when the strand is completed the shoe is turned 90 deg. and the loops are in a vertical plane. On this bridge the anchor shoes were left in the horizontal position. These departures from customary spinning practice were probably of minor significance.

The strands were spun, two at a time; strands Nos. 1 and 2 were spun first, then 3 and 4. Strand No. 5 was spun by itself, followed by 6 and 7. Each strand was a unit in itself, consisting of an endless loop carried 175 times around its two anchor shoes. The free end of the first wire placed in the strand was spliced to the back end of the last wire. The two sides of the loop were bound into a single strand about 15 ft. forward (toward the bridge) from the anchor shoe. Each wire, as it was placed around the anchor shoe was pulled up so that its sag in the catenary of the main span had a predetermined relation to that of a guide wire. Further provision was made for moving the anchor shoe forward or backward on the eye-bar by means of shims (shown in Fig. 10), so that the sag in the strand as a whole could be adjusted to its proper position in the cable. These adjustments were designed to equalize the load on the cable among all of the wires composing it. The radius of curvature around the base of the anchor shoe was $9\frac{3}{4}$ in. The curvature over the saddle at the cable bent was 14 ft. and that over the saddle on the main tower was 10 ft. The individual strands were brought together about 18 ft. forward of the anchor shoes, where they were compacted into the circular section of the cable. A splay casting encircled the cable at this point. It is possibly of some significance that the wires were bent on a smaller radius of curvature where they entered the splay casting than where the cable passed over the saddles at the cable bent and main tower.

There was one innovation in the stringing of the wires for the cables of the Mt. Hope bridge that was one of the factors determining the stress con-

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[10] "The Hudson River Suspension Bridge," *Mechanical Engineering*, Vol. 52, No. 6, p. 587 (1930).

[11] E. E. Thum, "High Strength Wire for Suspension Bridges, Its Use and Abuse," *Metal Progress*, Vol. XXI, No. 6, June, 1932, p. 46.

ditions that caused the failures in the wire on the bridge, as is shown in a later portion of this paper. When the heat-treated wire was unwound from the 5-ft. diameter coils, at the heat-treating unit, or from the 7-ft. shipping reels, it would lie straight. It was recognized that in bending around the circular portion of the anchor shoe, 19½ in. in diameter, this wire would be stressed beyond its yield point. Consequently, when bent and then held to the curvature of the anchor shoe, from its originally straight form, the wire would be under elastic bending stresses equal, at the outer fibers, to the elastic limit which was known to be high. The bridge was designed so that the tensile stress in the wire, under maximum load on the cables, would be not over 85,000 lb. per sq. in., about one-half of the stress necessary to produce permanent set in the wire in a tension test. The margin of safety would be decidedly decreased with high elastic bending stresses in the wire at the anchor shoes. To avoid this circumstance, it was decided to preform each wire, at the loop around the anchor shoe, by bending it 1¼ turns (540 deg.) around a sheave 9½ in. in diameter. When released from this preforming sheave, the loop would spring open, because of the high elastic limit of the wire, to the curvature of the circular portion of the anchor shoe (19½ in. in diameter). The loop was then placed around the shoe where it would "lie dead," and be subjected only to tensile stresses. Preforming sheaves are mounted on the anchor shoes, their position relative to the tangent point of the shoe being indicated in Fig. 10.

It is at once apparent that a considerable degree of skill and care was necessary to obtain the preformed curvature *exactly* in that portion of the wire that was finally placed in contact with the circular portion of the shoe. It was stated that two men were assigned to each anchorage and that all of the wire at each anchorage was placed on the shoes by the two men assigned there originally. The extent to which the free curvature of the wires, after the cables were dismantled, matched the curvature of the anchor shoes from which they were removed, is shown in a later portion of this paper.

FAILURE OF THE WIRE (GENERAL CONSIDERATIONS)

Spinning of the original cables on the Mt. Hope bridge was begun on September 10 and completed on October 25, 1928. Construction of the bridge proceeded according to program and had progressed to the laying of the floor structure when, early in February, 1929, a few broken wires were observed at one of the anchorages. They were scattered among a number of the anchor shoes. On February 22, after a snow storm, 65 broken wires were discovered in one of the strands, and on the following day, an additional number. All of the fractures had occurred at or close to the tangent point on the anchor shoe.

The broken wires were repaired by pulling out the broken ends and splicing in, some distance forward of the anchor shoe, a "hairpin" of cold-drawn wire. The heat-treated wires were breaking faster than repairs could be made and drastic steps were believed to be necessary to prevent collapse of the bridge. Hold-back yokes were fastened to each end of each cable and tied back to the anchorages to take up a portion of the load from the cables. Breakages practically ceased after this was done. It was, of course, evident that the heat-treated wire was unsafe and that the bridge had to be dismantled at once.

TABLE V.—NUMBER OF BROKEN WIRES IN EACH STRAND.

Strand	East Cable			West Cable		
	Bristol Anchorage	Portsmouth Anchorage	Total	Bristol Anchorage	Portsmouth Anchorage	Total
No. 1	140	4	144	4	6	10
No. 2	6	10	16	3	2	5
No. 3	7	16	23	10	129	139
No. 4	4	11	15	3	0	3
No. 5	2	3	5	1	1	2
No. 6	2	22	24	6	1	7
No. 7	2	2	4	3	2	5
			231			171

Total, both cables—402.

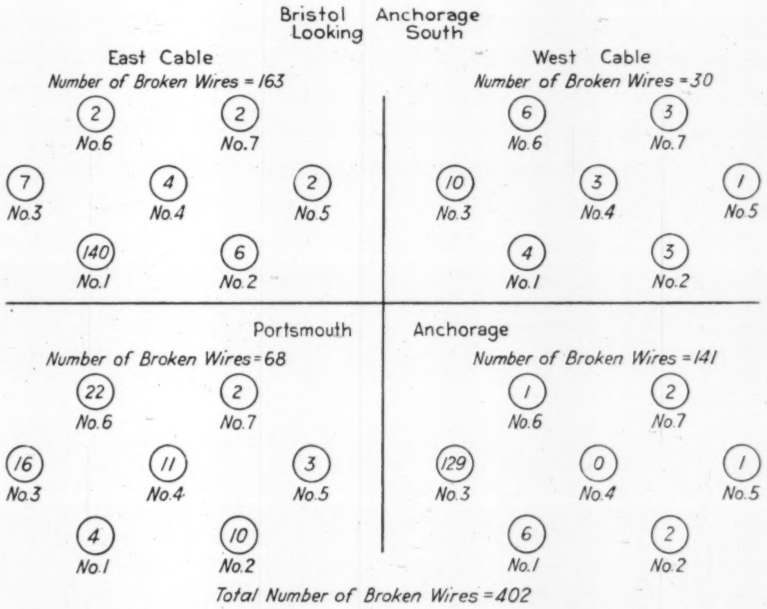
Bristol Anchorage
Looking South
East Cable
Number of Broken Wires = 163
2
No.6
2
No.7
7
No.3
4
No.4
2
No.5
140
No.1
6
No.2
West Cable
Number of Broken Wires = 30
6
No.6
3
No.7
10
No.3
3
No.4
1
No.5
4
No.1
3
No.2
Portsmouth Anchorage
Number of Broken Wires = 68
22
No.6
2
No.7
16
No.3
11
No.4
3
No.5
4
No.1
10
No.2
Number of Broken Wires = 141
1
No.6
2
No.7
129
No.3
0
No.4
1
No.5
6
No.1
2
No.2
Total Number of Broken Wires = 402

FIG. 11.—Distribution of Breaks in Wire at Anchor Shoes, Mt. Hope Bridge. Numbers in circles indicate broken wires.

Construction at the Ambassador bridge had not progressed quite so far, but breakages of the same kind appeared there and that bridge also was dismantled.

In dismantling the cables of the Mt. Hope bridge, comparatively few broken wires were found in the cables at locations between the anchorages. Approximately 20 were found under the saddle clamps at the cable bents. None was found at the main tower saddles; eleven were found under cable bands in the side and main spans. The cable bands were heavy steel castings encircling the cables, over which were looped the suspender ropes attached to the floor structure. Most of these breaks were at the outside of the cables and were believed to have occurred at points of localized stress where the saddle covers or bands had pinched the wire. These breaks probably would not have been discovered had the bridge not been dismantled. Their number, 31, was but a small fraction of the total number of 4900 parallel wires in the two cables.

The total number of wires that had broken at the anchor shoes when the cables were dismantled was 402, distributed as shown in Table V and Fig. 11. As each wire broken at the anchor shoe left two loose ends for the wires in the cables, the breaks amounted to 804 loose ends in the 4972 parallel wires in the two cables, approximately 16 per cent. A few instances were observed where two breaks had occurred in the same wire at opposite sides of the anchor shoe.

A striking fact was that 140 broken wires (in a total of 175) were in strand No. 1 at the Bristol anchorage of the east cable, and 129 were in strand No. 3 at the Portsmouth anchorage of the west cable. At the opposite ends of these two strands there were, respectively, 4 and 10 broken wires.

The dismantling of the cables was done in the presence of representatives of the National Bureau of Standards, who supervised the collection of the material sent to the Bureau for this study. Each strand was cut 20 to 30 ft. forward from the anchor shoe and the portion including the loops around the shoe was shipped to the Bureau intact. Unfortunately, many of the broken ends had been removed from the loops when repairs had been made, and were not shipped with the loops. However, the broken ends which remained were believed to represent accurately the types of fracture that occurred in all of the broken wires. In addition, 19 wires, 700 ft. long, were obtained from a strand in one of the side spans, together with other random lengths from various portions of both cables.

In discussions at the time the Bureau entered this investigation, a number of theories were advanced to account for the failure of the wire, and many helpful suggestions were received.

Among others, some of the conjectures at the time were:

1. *Segregated Steel.*—It was thought that possibly ingotism had not been obliterated because of insufficient reduction by mechanical working from ingot to wire. Furthermore, if segregated billets had escaped detec-

tion, occasional coils of wire might have failed to respond to the heat treatment, or contained weak spots as a result of inhomogeneity in composition.

2. *Unsuitable Steel.*—Traditionally, acid open-hearth steel has been considered as of higher "quality" than basic steel. The steel used for the conventional cold-drawn bridge wire has been made, almost without exception, in acid furnaces. The steel used for the heat-treated wire had been made in basic open-hearth furnaces. Because of this fact, undiscovered accidental variations in "quality" or the use of billets from a faulty heat were considered as possible explanations for the large number of broken wires in two of the strands.

3. *Defective Heat Treatment and Unreliability of Heat-Treated Steels.*—In the section on "Manufacture of the Heat-Treated Wire," evidence was presented to show that whatever properties were imparted to the wire by the heat treatment, they remained fixed as they were when the wire left the heat-treating unit. The mere fact of the failure is sufficient evidence that the properties, as produced, were not adequate to withstand the service conditions imposed on the wire. Whether or not the failure could have been avoided by a different heat treatment of the same steel is a matter for further discussion.

4. *Abuse of the Wire During Erection.*—The cables of the Mt. Hope and Ambassador bridges were erected by the same construction company that had previously erected the cables on the Philadelphia-Camden bridge, and which subsequently spun the cold-drawn wire into the cables when the first-named bridges were re-erected. In so far as the spinning of the original cables followed conventional procedure, it was done by skilled workmen with previous experience. It was learned, however, that the shaping of the wire to fit, even approximately, the curvature of the anchor shoes had not been accomplished in the preforming operation, as is shown later in this paper.

It can be shown readily, without resorting to data from laboratory investigations of the failed wire, that several of these conjectures cannot be reconciled with known facts.

The 402 breaks for which an explanation was sought were confined strictly to the loops around the anchor shoes and were located almost without exception at or close to the tangent point between wire and shoe. Breaks had occurred at all but one of the 28 anchor shoes. Accepting the calculations of the bridge engineers, the nominal static load on the cables at the time the failures occurred produced a tensile stress in the wire of approximately 32,000 lb. per sq. in., about one-seventh of the ultimate strength of the wire. If, because of segregation, the wire contained localized points of weakness that caused failures under a static tensile stress of this magnitude, it is entirely illogical to suppose that such points of weakness could have been localized entirely at the anchorages. In all of the studies made at this Bureau, the material in specimens of the fractured wires, taken close to the points of fracture, could not be distinguished, physically or chemically, from that in specimens of unfractured wires, or of fractured wires some distance removed from the point of failure. The logical conclusion is that the stress conditions at the anchor shoes were radically different from those in the compacted cables. Segregation in the steel was given no further consideration as a cause of the failure of the wire.

It was found, as shown in Table V and Fig. 11, that 144 fractures had occurred at the two anchor shoes of strand No. 1 of the east cable, 140 of the breaks at the Bristol anchorage and only 4 at the Portsmouth anchorage. In No. 3 strand of the west cable 129 breaks had occurred at the Portsmouth anchorage but only 10 at the Bristol anchorage. In the total of 402 breaks at the 28 anchor shoes, 283 had occurred in two of the strands, 269 of them at two of the anchor shoes. This fact might be supposed to indicate that these two strands were composed largely of wire from one or two bad heats of steel. Several facts indicate that this was extremely improbable.

The two cables of the Mt. Hope bridge were composed of 5132 coils of wire from 55 heats of steel. The identity of each coil and the heat number from which it was produced were maintained up to the time the wire was transferred from reel to cable strand. After the wire was placed in the strand this identity was lost. Strand No. 1 of the east cable contained 360 coils from 27 heats, spun into 177 complete loops around two anchor shoes or 354 parallel wires in the bound strand. Strand No. 3 of the west cable contained 360 coils, from 24 heats, spun into 176 complete loops, or 352 parallel wires in the strand. Wire from 21 heats was present in both strands. It is highly improbable that two-thirds of the total number of breaks in both cables were caused by the chance location, at 2 of the 28 anchor shoes, of wire from one or more bad heats of steel. The evidence does indicate that at these 2 anchor shoes there must have been a condition differing systematically from the conditions at all of the other shoes. No such differences were observed in the anchor shoes themselves and the equal load distribution was not questioned. If, however, the preforming sheaves were located in a different position, relative to the tangent points on these two anchor shoes, from their positions on the other 26 anchor shoes, this circumstance would have caused a systematic difference in the stress conditions in the wire at the points where the fractures occurred. The preforming sheaves were removed after the cables had been spun, and their exact location on any of the shoes could not be ascertained after the failures occurred.

5. *Hydrogen Embrittlement.*—For the same reason that the presence, in the Mt. Hope bridge cables, of wire from one or more bad heats of steel could not be reconciled with the distribution of the fractures, hydrogen embrittlement of the steel as a probable cause of the failure was also discarded from further consideration.

6. *Acid versus Basic Open-Hearth Steel.*—The carbon, phosphorus and sulfur contents of the failed wire conformed to the composition requirements specified for the acid open-hearth steel used in the cable wire that replaced the failed wire on the Mt. Hope and Ambassador bridges, and in the cables of the Hudson River and Delaware River bridges. Whether or not acid open-hearth steel of the same composition as the basic steel would have acquired the same physical properties after having been processed in the same way as the failed wire is an academic question.

In so far as the final structure and properties were dependent upon the carbon content, the same results would be expected. In a number of inclusions, the acid steel used in the replacement wire on the Mt. Hope bridge could not be distinguished from the basic steel of the heat-treated wire, as is shown in Figs. 12 and 13. Evidence has not been produced that the wire, in the physical condition in which it was placed in the cables, would not have failed under the service conditions imposed upon it, regardless of whether the steel was produced in acid or basic open-hearth furnaces. The use of basic open-hearth steel was given no further consideration as a determining factor in the failure of the heat-treated wire.[12, 13]

FIG. 12.—Inclusions in Specimen of Wire from Mt. Hope Bridge. Basic open-hearth steel. Unetched (× 100).

FIG. 13.—Inclusions in Specimen of Cold-Drawn Bridge Wire. Acid open-hearth steel. Unetched (× 100).

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[12] An excellent discussion of the various factors concerned with the problem of the failure of the heat-treated wire, based on information available at the time, was presented by E. E. Thum, Editor, in *Metal Progress*, Vol. XXI, No. 6, June, 1932; Vol. XXII, Nos. 1, 2 and 3, July, August, and September, 1932.

[13] Other details, concerning the design and construction, failure of the wires, and dismantling of the Mt. Hope and Ambassador bridges, not given in this paper, were presented by *Engineering News-Record* in the following articles:
Design of Mt. Hope Wire-Cable Suspension Bridge, Vol. 100, p. 585 (1928).
Design of Great International Suspension Bridge over Detroit River, Vol. 101, p. 460 (1928).
Broken Wires Cause Rejection of Cables on Mt. Hope Suspension Bridge, Vol. 102, p. 516 (1929).
Cables on Detroit River Suspension Bridge to Be Replaced, Vol. 102, p. 564 (1929).
Technical Aspects of Cable Wire Breakages on the Mt. Hope Suspension Bridge, Vol. 102, p. 602 (1929).
Dismantling Two Long Suspension Bridges, Vol. 103, p. 562 (1929).

LABORATORY STUDIES OF THE HEAT-TREATED WIRE

An obvious point of attack in the investigation of the material sent to the National Bureau of Standards from the Mt. Hope bridge was to make direct comparisons of the mechanical properties of wires which had broken and those which had not broken at the anchor shoes.

Tensile Properties:

In the course of the investigation, the ordinary tensile properties were determined on several hundred specimens from wires in both categories. The results are summarized in Table VI.

The differences between specimens from wires that had broken and specimens from unbroken wires were no greater than the differences between wires from either group. Many of the specimens were taken from the preformed loop around the anchor shoes, and adjacent to the original fractures in the broken wires. With the exception of low values for reduction of area at the point of fracture, the results of the tension tests made after the failure were in good agreement with the specified requirements. Evidently the tensile properties had not been altered by preforming or by service on the bridge.

TABLE VI.—TENSILE PROPERTIES AND HARDNESS OF HEAT-TREATED AND COLD-DRAWN WIRE.

	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.[a]	Elongation in 4 in., per cent	Reduction of Area, per cent	Rockwell Hardness, "C" Scale	Vickers Hardness, 30-kg. Load
HEAT-TREATED WIRE FROM BRIDGE						
Average	223 000 ± 5 000[b] (223 specimens)	189 800	6.1 ± 0.8[b] (13 specimens)	37 ± 9[b] (220 specimens)	46 ± 1[b] (190 specimens)	486 ± 6[b] (90 specimens)
Maximum	237 000	196 200	7.5	53	50	511
Minimum	201 000	180 100	5.0	9	43	454
COLD-DRAWN WIRE REPLACEMENT						
Average	223 000 ± 2 500[b] (16 specimens)	182 600	6.6 ± 0.3[b] (16 specimens)	40 ± 2[b] (16 specimens)	44 ± 1[b] (75 specimens)	441 ± 4[b] (75 specimens)
Maximum	226 000	193 300	7.4	42	46	459
Minimum	216 000	178 200	6.4	33	39	420

[a] Yield point = stress to produce extension of 0.0075 in. per inch under load.
[b] ± values denote average deviation from mean.

The specified value for reduction of area was 30 per cent. According to the results shown in Table VI, the average for several hundred specimens from the bridge was 37 per cent with an average deviation from the mean of ± 9 per cent. In any length of wire, either from the loop around the anchor shoe or from a straight portion, it was found that, generally, one out of 7 to 10 tension test specimens fractured with practically no reduction of area. The tensile strengths of specimens with abnormally low reductions of area were, in all cases, within the range for specimens with normal reductions of area. Very seldom did adjacent specimens from the same piece have abnormally low reduction of area. This explains the fact that such wire was not rejected by the inspectors at the production plant. When a specimen from the end of a coil broke with low reduction of area, a retest was allowed and the second specimen, in practically all cases, had the required reduction of area.

No differences in microstructure or hardness of the steel were discovered between any specimen with low reduction of area and those of normal ductility. It was found that the cause lay in the surface conditions of the wire. A group of wires was assembled in which the percentage of specimens fracturing with low reduction of area was higher than average. When the zinc coating was removed with acid, the percentage of low reduction breaks was decreased appreciably as shown in Table VII. When the surface of the steel was polished after stripping off the zinc, the occurrence of low reduction breaks was almost eliminated. When the outer layers of the galvanized wire were removed mechanically to a depth of 0.015 in., all low reduction breaks were eliminated. All reductions of area less than 20 per cent were included as "low," in Table VII. In 93 per cent of the specimens counted in the low column, the reduction of area was less than 12 per cent. Because it could not be ascertained beforehand that a certain number of low reduction breaks would be obtained in a given number of specimens, there is some uncertainty to ascribing all of the decrease in percentages of low-reduction breaks to the various surface treatments listed in Table VII. The difference between the wire with the original surface intact and the wire reduced to 0.165 in. in diameter is sufficiently marked to conclude that low reduction of area at the fracture in a tension test was caused by defects in the outer layers of the wire, no deeper than 0.015 in. from the surface. In a tension test certain of these defects modified the mode and degree of

TABLE VII.—EFFECT OF SURFACE TREATMENT ON THE REDUCTION OF AREA IN TENSION TESTS OF THE HEAT-TREATED WIRE.

Condition of Specimen	Number of Specimens	Specimens with Low Reduction of Area	
		Number	Per cent
With original surface	60	28	46.7
Coating removed with acid	48	9	18.7
Coating dissolved off, steel surface polished	36	1	3.0
Galvanized surface polished, diameter reduced:			
from 0.195 to 0.193 in	13	4	30.7
from 0.195 to 0.190 in	13	4	30.7
from 0.195 to 0.188 in	10	3	30.0
Diameter reduced to 0.165 in.	32	0	0

deformation at the point of fracture. It is noteworthy also that removal of only the shallower defects did not cause any marked improvement. The decrease in ductility was evidently caused by the more deeply seated defects, occurring sporadically, but apparently in all of the wire. Their effect upon the fractures occurring on the bridge was manifested in a different manner.

Hardness Tests:

Rockwell hardness "C" scale and Vickers hardness were, within experimental error, uniformly the same throughout the section of any one specimen, and not significantly different in specimens selected at random from broken and unbroken wires from all of the strands. Typical results are given in Table VI.

TABLE VIII.—RESULTS OF TORSION TESTS ON BRIDGE WIRE.

MATERIAL	NUMBER OF SPECIMENS	NUMBER OF 360-DEG. TURNS, AVERAGE
Heat-treated wire:		
Original surface intact	55	11¼
Zinc coating removed with acid	33	17½
Zinc coating removed mechanically	5	15½
Drawn wire (1-hole):		
Not heat treated or galvanized	9	9½
Cold-drawn bridge wire:		
Galvanized	10	5¾
Zinc coating removed with acid	10	8¾

Torsion Tests:

As a possible means of detecting differences between the wires that broke on the bridge and unbroken wires, torsion tests were made on specimens from both groups. The length of the specimen between the grips of the torsion machine was 15 in. The number of twists or complete turns of 360 deg., before fracture, differed as much between specimens of one group as between specimens from the two groups. A small but distinct increase in number of twists was noted for specimens from which the zinc coating had been removed by solution in acid. It was significant, but not unexpected, that the drawn wire, not heat treated, withstood a lower number of twists than the heat-treated galvanized wire. The average number of twists for each condition of the wire are listed, to the nearest quarter turn, in Table VIII. For purpose of comparison, the results obtained with cold-drawn bridge wire, both galvanized and stripped, are also given.

The appearance of the torsional fractures was distinctly different in the two types of wire. In the heat-treated wire the fractured surfaces were smooth and flat, on a plane perpendicular to the axis of the specimen. In the cold-drawn wire the fractured ends were split longitudinally, no doubt as a result of the quasi-fibrous structure of this wire.

Coiling Experiments:

It was stated previously that if the wire, as it was taken from the processing unit, was wound on 30-in. diameter coils many breaks occurred shortly after the coils were stored. If the cause of such breaks was inherently a weakness of the steel in resisting elastic bending stresses and persisted to the time the failures occurred on the bridge, it is reasonable to expect that the wire recovered from the bridge would behave in a similar manner. Several thousand feet of wire from straight lengths, recovered from one of the side spans, were wound, in 1930, on 30-in. diameter coils. In the nearly six years since that time not a single break has occurred in any of these coils.

Because many steels are known to be less ductile at low temperatures than at ordinary atmospheric temperatures, it was thought that possibly, during the winter months immediately preceding the discovery of the fractures on the bridge, the wires might have become embrittled enough to cause them to snap off where the elastic bending stresses were highest, in the loops around the anchor shoes. About 1600 ft. of wire were wound on 19-in. diameter coils, some of it when at a temperature of approximately 5 F. The coils were alternately cooled to 5 F. for several hours, and then allowed to warm up to as much as 90 F. for the remainder of a day. This treatment was repeated over a period of about 2 months. Many of the coils, when at the low temperature, were dropped on a concrete floor. Not a single break occurred in any of these coils during the two-month period nor since that time. It was concluded that the wire when taken from the bridge was not subject to the brittle type of fracture as the result of static elastic bending stresses caused by holding it in 19 or 30-in. diameter coils.

These experiments, by themselves, do not disprove any of the conjectures made at the time the bridge was dismantled, namely, that some undetected change had occurred in the wire between the time the fractures had occurred on the bridge, early in 1929, and the time the studies were begun at the National Bureau of Standards. Evidence is presented later to answer this question.

Long-Time Loading of Loops Supported on an Anchor Shoe:

Theoretically, if the preforming operation accomplished its designed purpose, the wires in the loops around the anchor shoes were subjected only to tensile stresses arising from the pull of the cable on the anchorage. It was learned, however, upon examining the loops recovered from the bridge, that the wires, when constrained to the curvature of the anchor shoes by the pull of the cables, were subjected to elastic bending stresses as well. The behavior of the wire under a combination of tensile and elastic bending stresses was therefore studied.

Specimens of wire about 8 ft. long were subjected to static tensile loads while looped over a support that had the curvature of the circular portion of the anchor shoes on the bridge. The loading device is shown in Fig. 14. The specimen of wire, *H*, was carried in a broad flat groove over the top of the semi-circular metal casting, *C*, with a radius of curvature $9\frac{3}{4}$ in. Each end of the wire was gripped between two steel blocks *G* held together with bolts. The yoke *Y* rested on top of, and transmitted the load to, the grips *G*. A downward pull was exerted on the yoke by a simple lever system linked to the yoke at *L*. The knife edges K_1 and K_2, mounted in the same horizontal plane, and forming the fulcrum points of the lever, were of hardened steel about 1 in. square in section. These and the loops in which they operated were of the type used in railroad track scales. The ratio of the two lever arms was approximately 30 to 1. The weight of the long arm of the T-section beam *B* was sufficient to exert a force equivalent to a tensile stress of approximately 50,000 lb. per sq. in. in each leg of the wire *H*. By adding weights to the pan *W*, a pull of 13,000 lb. could be exerted at the link *L*, sufficient to fracture the wire specimens at a tensile strength of 220,000 lb. per sq. in. By means of counter weights, the effective weight of the beam *B* was decreased when stresses less than 50,000 lb. per sq. in. were desired. The machine was calibrated for tensile loads exerted on the link *L* up to 6000 lb. by means of a Morehouse proving ring. Five of these units were constructed.

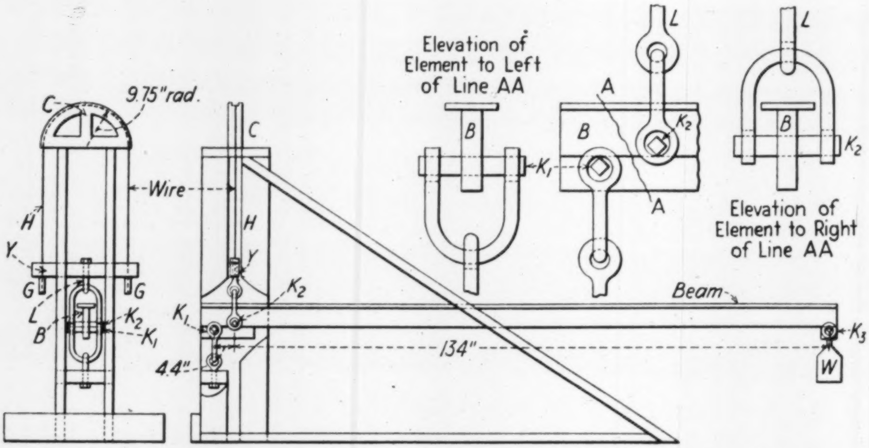


FIG. 14.—Device for Subjecting Specimens of Bridge Wire to Tensile Loads.

Specimens of the heat-treated wire from the bridge were subjected to an initial load equivalent to a tensile stress of 90,000 lb. per sq. in. In some cases this load was not changed for 30 days, after which the load was increased each day by an increment corresponding to 10,000 lb. per sq. in.

until the wire broke. In other cases the initial load was held for only one day and then increased in the same manner. Some of the specimens were not preformed, others either had been preformed at the bridge or were preformed in a similar manner in the laboratory. In addition, some were preformed on a sheave 4 in. in diameter. The laboratory preforming was done at both 68 F. and at freezing temperatures, 10 to 32 F. In some cases the center of the preformed loop was placed at the midpoint of the curved support, in others it was intentionally displaced to one or the other side of the midpoint. Similar tests were made on heat-treated wire stripped of its zinc coating and on cold-drawn galvanized bridge wire. The latter wire normally had a radius of curvature of 40 in. All of the specimens when relieved of the tensile load deviated from the curvature of the support, and in practically all cases by different amounts.

TABLE IX.—TESTS UNDER COMBINED TENSILE AND BENDING STRESSES. TENSILE LOADS HELD CONSTANT FOR VARIOUS PERIODS.

Shape of Specimen, unloaded	Loading Condition[a]	Heat-Treated Wire							Cold-Drawn Wire	
		Galvanized					Stripped		Galvanized	
				Fractured at Low Tensile Strength						
		Number of Specimens	Fractured at Nominal Tensile Strength (220,000 lb. per sq. in.)	Number	Stress, lb. per sq. in.	Fractures with Low Reduction of Area (<0.20 per cent)	Number of Specimens	Fractures at Nominal Tensile Strength and Normal Reduction of Area	Number of Specimens	Fractures at Nominal Tensile Strength 220,000 lb. per sq. in. and Normal Reduction of Area
Straight	A	7	7	0	....	3	..	..	..	..
Preformed on 9½-in. sheave at 68 F.; mid-point of curvature of wire and shoe coincided	A	3	3	0	....	2	..	..	..	..
Preformed on 9½-in. sheave at 68 F.; mid-point of curvature in wire displaced from that on shoe	A	10	10	0	....	5	..	..	..	..
Straight	B	7	5	2	160 000 160 000	2	3	3	5	4
Preformed on 9½-in. sheave at 68 F.; mid-point of curvature of wire and shoe coincided	B	6	6	0	....	2	1	1	1	1
Preformed on 9½-in. sheave at 68 F.; mid-point of curvature in wire displaced from that on shoe	B	12	10	2	170 000 150 000	5	1	1	..	..
Preformed on 4-in. sheave at 68 F	B	7	0	0	....	1	2	2	...	..
Preformed on bridge	B	7	0	0	....	0	..	..	..	..
Preformed on 9½-in. sheave at 32 F. or lower	B	36	32	4	120 000 130 000 190 000 195 000	7	6	6	4	4
Preformed on 4-in. sheave at 32 F. or lower	B	40	40	0	....	7	7	7	5	5

[a] Loading Conditions A: Initial tensile stress 90,000 lb. per sq. in., held 30 days then increased 10,000 lb. per sq. in., daily to fracture.

Loading Conditions B: Initial tensile stress 90,000 lb. per sq. in., increased daily 10,000 lb. per sq. in. to fracture.

The results of the tests are shown in Table IX. Of the 135 specimens of the heat-treated galvanized wire, only 8 broke at tensile stresses less than the nominal tensile strength, and none at a tensile stress less than 120,000 lb. per sq. in. In many of the specimens, the curvature, unloaded, deviated from that of the support by far greater amounts than the unbroken loops from strands recovered from the bridge. Apparently the elastic bending stresses had not much effect in lowering the effective resistance of the specimens to static tensile loads. It is possibly significant that the fractures in the 8 specimens that broke at less than nominal tensile strength all occurred in the portion of the specimen in contact with the support. Thirty-four of this group, including the eight "low-tensile" breaks, broke with less than 20 per cent reduction of area at the point of fracture. The bending stresses acting in conjunction with the tensile stresses evidently did tend to cause a higher than normal proportion of the brittle type of fracture. Possibly some of the specimens had been damaged to a greater extent by preforming in the laboratory than was the case with the wires on the bridge.

Of the 20 specimens with the zinc coating removed by solution in acid, all fractured with normal reduction of area when the tensile stresses reached the nominal tensile strength. The same was true for the 15 specimens of the cold-drawn galvanized bridge wire.

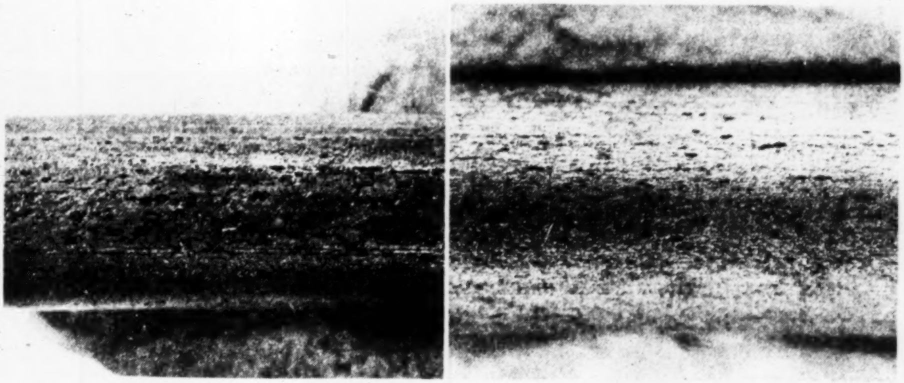
The wires at the anchor shoes on the bridge were subjected to a combination of bending and tensile stresses. The bending stresses necessarily were within the elastic range, but in many cases, as shown later, were equal to the elastic limit. According to the calculations of the bridge engineers the tensile stresses had not exceeded 32,000 lb. per sq. in. In the laboratory tests just described many of the specimens were subjected to elastic bending stresses equal to the elastic limit, yet no failures were obtained under combined stresses until the static tensile stress was at least four times as great as the reported tensile stress in the cable wires at the time of failure. Although the tensile loads were increased at a slow rate, the increases were made by increments, and the stresses, between increments, were static. It would be contrary to experience in testing of metals to expect significantly different results merely by making the increments of tensile load smaller, even to the point of infinitesimal smallness, so long as the load was *continuously* increased. The results of the static loading under combined stresses do not furnish an adequate explanation of the mechanism of failure of the wire on the bridge.

Fatigue Tests:

The studies so far described indicated that the wire recovered from the bridge was essentially the same metallographically and in its mechanical properties as at the time it was accepted for use on the bridge. Its strength was adequate to resist fracture under static stresses much higher than the calculated stresses at the points of failure. The fact that the wire did fail suggested that a study should be made of the resistance of the wire to fluctuating stresses.

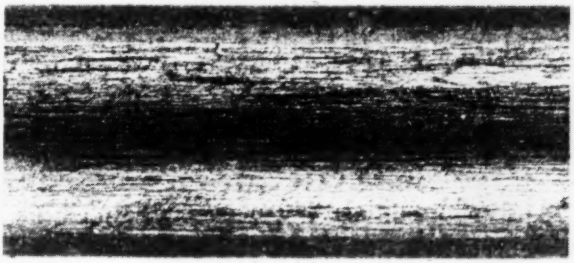
Results of extensive fatigue tests of the heat-treated wire and of cold-drawn bridge wire have already been published.[14,15,16]

The fatigue limit of the steel, determined by the rotating-beam method on specimens with polished surfaces, machined from the heat-treated wire, from an original diameter of 0.195 in. to a reduced diameter of 0.150 in., was 110,000 lb. per sq. in., approximately one-half the tensile strength. For the cold-drawn replacement wire, the fatigue limit determined in the same way was 118,000 lb. per sq. in., a value slightly but not significantly higher than that for the heat-treated steel. These values are the normal expectancy for steels having the physical properties in other respects possessed by the two wire steels.



(*a*) Heat-treated wire (× 5).

(*b*) Heat-treated wire (× 6).



(*c*) Cold-drawn wire (× 5½).

FIG. 15.—Appearance of Surface of Bridge Wire After Galvanized Coating Had Been Removed by Solution in Acid.

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[14] S. M. Shelton, "Fatigue Testing of Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 204 (1931).

[15] S. M. Shelton and W. H. Swanger, "Fatigue Tests of Galvanized Wire Under Pulsating Tensile Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 348 (1933).

[16] S. M. Shelton and W. H. Swanger, "Fatigue Properties of Steel Wire," National Bureau of Standards *Journal of Research*, Vol. 14, p. 17 (1935).

It had been ascertained that in the heat-treated wire the fractures that occurred at stresses below the nominal tensile strength, including those at the anchor shoes on the bridge, invariably originated at the surface. It was evident that conditions at the surface of the wire were involved. A method was devised[14] for making rotating-beam fatigue tests on specimens of the failed wire with the original surface intact. For specimens in this condition the fatigue limit was only 50,000 lb. per sq. in., which is less than half of that for the steel itself, with all damaging surface defects removed. For specimens from which the zinc coating had been removed by solution in acid, the slightly higher fatigue limit, 60,000 lb. per sq. in. was obtained.

These results showed that a drastic reduction in resistance to repeated stresses was caused by the characteristics of the surface of the heat-treated wire. As in the observations made on the causes for low reduction of area in tension tests, the removal of the zinc coating provided some improvement, but it required removal of the surface layers to a depth below the bottom of the worst defects to effect a real improvement. Examinations of numerous specimens disclosed ample evidence that surface defects similar to those shown in Fig. 15(*a*) and (*b*) existed throughout all of the heat-treated wire.

Except for the decrease in the amount of reduction of area at the point of fracture in a tension test, such defects did not, in general, affect the other tensile properties. It is possible that some of the fractures obtained in tension tests at less than the nominal strength were the result of surface defects large enough to cause a decrease in effective cross-sectional area. That such surface defects cause a marked lowering of the endurance strength of steels, and especially of high-strength steels, has been demonstrated in many reports of investigations of the subject.

In the case of the heat-treated wire, the pits were undoubtedly formed where the effects of mill scale or other rolling defects had not been ironed out in the one or two drafts the wire received in drawing. The scale was finally removed and the pits possibly enlarged in the pickling baths preceding the galvanizing operation, and then filled with zinc in the last operation. The examples shown in Fig. 16 are typical illustrations. It is well known that in a hot-dip galvanized coating the layer next to the steel consists largely of brittle iron-zinc compounds. The filling in the pits consisted largely of this material.

The pits themselves served as points of localized stress concentration. Moreover, the fatigue strength of the zinc and particularly of the iron-zinc compounds is much lower than that of the steel. Cracks were readily formed in the zinc coating at stresses much less than the fatigue limit of the steel. These cracks, once formed, constituted in themselves points of localized stress concentration and were readily extended into the steel under the action of the repeated stresses in the fatigue tests. Numerous instances were found of cracks in the iron-zinc alloy extending into the steel beneath.

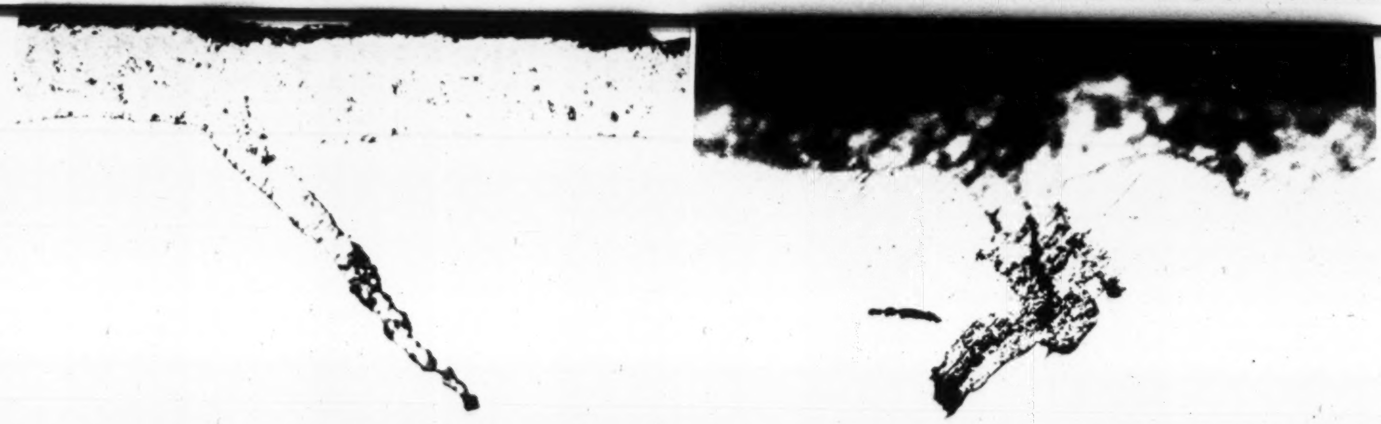

FIG. 16.—Pits in Heat-Treated Wire, Filled with Zinc (× 500).
Longitudinal sections. Galvanized coating at top of micrograph.

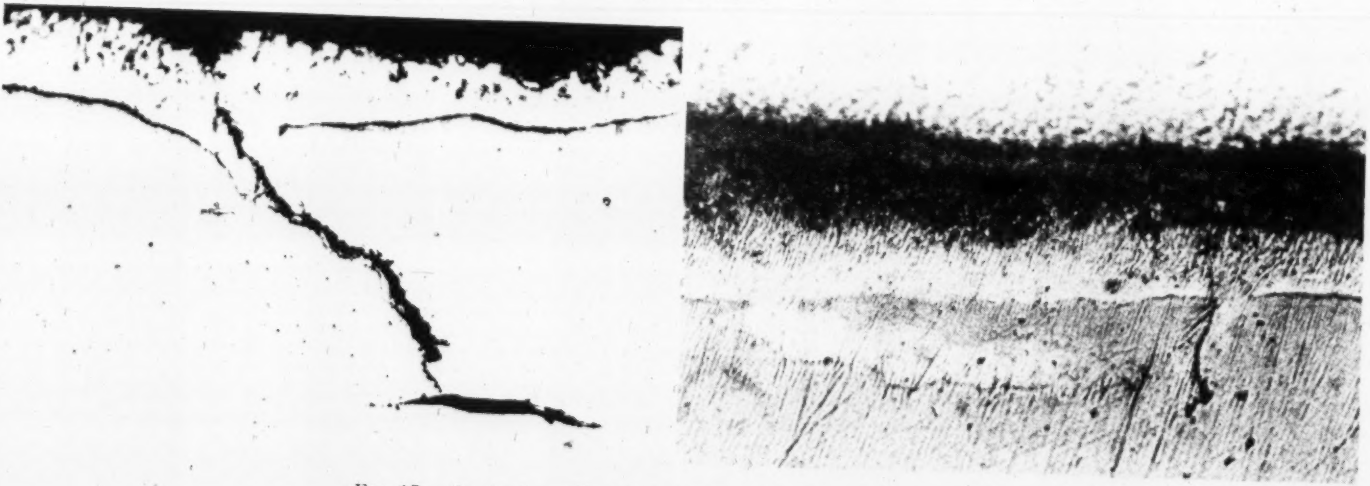

FIG. 17.—Cracks from Zinc Coating Extending into Steel (× 500).
Heat treated wire. Longitudinal sections.

Typical examples are shown in Fig. 17. With the zinc coating removed before the fatigue test was begun, it required only a slight increase in maximum fiber stress to start a fatigue crack at the point of maximum stress concentration at the bottom of a favorably located pit.

The cold-drawn wire, drawn through five dies from hot-rolled rod, had a much smoother surface with comparatively few pits, as shown by a typical example, Fig. 15(*c*). The cold-drawn wire had a permanent set on a radius of curvature of 40 in. which could not be removed without altering its physical properties. Because of this curvature, specimens could not be rotated smoothly and fatigue tests by the rotating-beam method could not be made on specimens of this wire with the original galvanized surface intact, nor with the zinc coating removed by solution in acid.

TABLE X.—RESULTS OF PULSATING TENSION AND ROTATING-BEAM FATIGUE TESTS.
Each range was the maximum permitting 10,000,000 cycles of repeated stress without fracture.

Condition	Heat-Treated Wire				Cold-Drawn Wire			
	Mean Stress, lb. per sq. in.	Maximum, lb. per sq. in.	Minimum, lb. per sq. in.	Range, lb. per sq. in.	Mean Stress, lb. per sq. in.	Maximum, lb. per sq. in.	Minimum, lb. per sq. in.	Range, lb. per sq. in.
Galvanized.........	49 000	76 000	22 000	54 000	50 000	80 000	20 000	60 000
	68 000	92 000	44 000	48 000	73 000	97 000	49 000	48 000
	88 000	111 000	65 000	46 000	90 000	114 000	66 000	48 000
	107 000	130 000	84 000	46 000	110 000	135 000	85 000	50 000
	133 000	158 000	108 000	50 000	135 000	156 000	114 000	42 000
	153 000	177 000	129 000	48 000	156 000	178 000	134 000	44 000
	0[a]	50 000	−50 000	100 000	....	....	......	....
Stripped...........	50 000	80 000	20 000	60 000	50 000	80 000	20 000	60 000
	75 000	100 000	50 000	50 000	75 000	103 000	47 000	56 000
	100 000	126 000	74 000	52 000	100 000	128 000	72 000	56 000
	0[a]	60 000	−60 000	120 000	....	....	.....	....
Machined to 0.150-in. diameter......	85 000	143 000	27 000	116 000	85 000	155 000	15 000	140 000
	100 000	156 000	44 000	112 000	100 000	165 000	35 000	130 000
	125 000	164 000	86 000	78 000	125 000	188 000	62 000	126 000
	0[a]	110 000	−110 000	220 000	0[a]	118 000	−118 000	236 000

[a] Rotating-beam tests.

Since the wires in suspension bridge cables are tensile members, stresses that cause fatigue fractures can arise only from fluctuation in tensile load. The wires would not be expected to be subjected to complete reversals of stress as in a rotating-beam fatigue test. It was considered desirable, therefore, to study the behavior of the bridge wire under pulsating tensile stresses. After suitable gripping devices were developed[16] for holding specimens in the Haigh alternating stress testing machine, determinations were made of the limiting ranges of tensile stress for various mean stresses. Determinations were made on both heat-treated and cold-drawn wire recovered from the bridge,[17] on specimens with the original surface intact, on specimens with the zinc coating removed by solution in acid,[18] and on specimens machined to a reduced diameter of approximately 0.150 in. in the middle of the gage length.

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[17] The cold-drawn wire recovered from the bridge consisted of the "hairpins" used to repair the breaks in the heat-treated wire, before the bridge was dismantled. It was similar to the wire used when the bridge was rebuilt.

[18] To remove the zinc coatings the specimens were immersed in hydrochloric acid (sp. gr. 1.19) to which had been added 1.6 g. antimony chloride per liter of acid: A.S.T.M. Standard Methods of Determining Weight and Uniformity of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (A 90 – 33), 1933 Book A.S.T.M. Standards, Part I, p. 317.

The results are listed in Table X together with the results of the rotating-beam tests, and are also plotted on the curves in Fig. 18.

For both types of wire, with a galvanized coating, the safe range of pulsating tensile stress for 10,000,000 cycles without fracture was approximately the same at all mean stresses employed. With the zinc coating removed, only slightly greater ranges were obtained in both types of wire, at the three mean stresses employed. With specimens machined to a diameter of 0.150 in. in the middle of the gage length (radius of curvature on the reduced section, 3 in.) the range for the cold-drawn wire was more than doubled although the range decreased slightly as the mean stress was increased. In general the same was true for the heat-treated wire with a machined and polished reduced section (0.150 in. in diameter), although the safe range of pulsating tensile stress was not as great and decreased more rapidly with increase in mean stress.

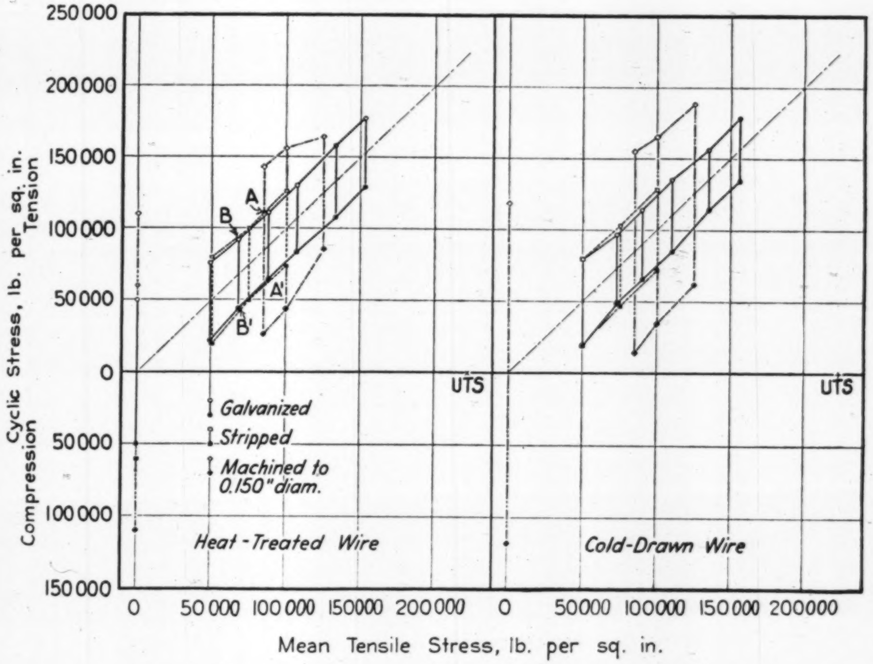
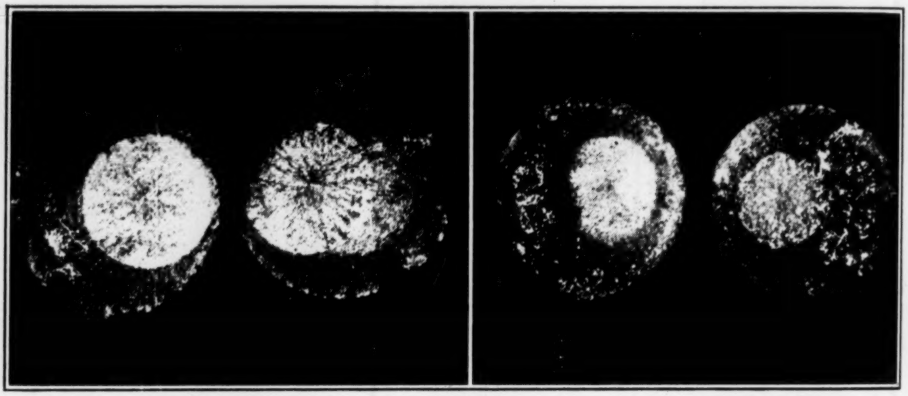


FIG. 18.—Results of Pulsating-Tensile-Stress and Rotating-Beam Fatigue Tests on Heat-Treated Wire from Mt. Hope Bridge and Cold-Drawn Replacement Wire.

Fatigue limits based on 10,000,000 cycles of repeated stress without fracture.

Even though the tensile strength of each type of wire was approximately 220,000 lb. per sq. in., the maximum range of pulsating tensile stress, at mean stresses from 50,000 to 150,000 lb. per sq. in. was only approximately 50,000 lb. per sq. in. for the galvanized wires, less than one-quarter of their ultimate strengths. Moreover, the heat-treated wire, with a much more severely pitted surface on the steel than the cold-drawn wire, had an endurance strength, both with and without the zinc coating, approximately as great as that of the cold-drawn wire.

It is important to note that the resistance of either type of wire to pulsating tensile stresses is determined, not by the magnitude of the maximum stress, but by the magnitude of the range of tensile stress. In other words, when subjected to repeated loading within the ultimate strength, the important question is not "How heavily is the wire loaded?" but "How far is it unloaded?"



(*a*) Heat-treated wire. (*b*) Cold-drawn wire.

FIG. 19.—Fractured Surfaces of Two Specimens of Bridge Wire (× 8).

Specimens machined from 0.195 to 0.150-in. diameter. Fractured in pulsating-tensile-stress fatigue tests. Note internal nucleus of fracture in each specimen.

This fact is readily demonstrated. For the limiting range $A - A'$ (Fig. 18, heat-treated wire) at the mean stress 88,000 lb. per sq. in., the maximum stress was 111,000 lb. per sq. in. and the minimum 65,000 lb. per sq. in. If the minimum stress were dropped to 25,000 lb. per sq. in., the maximum stress remaining unchanged, the mean stress would be 68,000 lb. per sq. in. at which the safe range $B - B'$ (Fig. 18) was determined. In determining this range ($B - B'$) one of the specimens broke after approximately 230,000 cycles of tensile stress between 102,000 and 34,000 lb. per sq. in. A break at still fewer cycles would be expected if it were stressed between 111,000 and 25,000 lb. per sq. in.

Of the many specimens of both types of wire broken in the fatigue tests, only two were obtained in which the nucleus of the fatigue fracture was in the interior of the section. The appearance of the fractured surfaces is shown in Fig. 19. Both were machined to a reduced diameter of slightly less than 0.150 in. in the mid-section, where the breaks occurred under pulsating tensile stress. Figure 19 (*a*) endured 800,000 cycles of tensile stress over the range 145,000 to 25,000 lb. per sq. in., and Fig. 19 (*b*), 900,000 cycles over the range 190,000 to 60,000 lb. per sq. in. Each was stressed at slightly more than the safe range determined at the mean stresses of 85,000 and 125,000 lb. per sq. in., respectively (Table X).

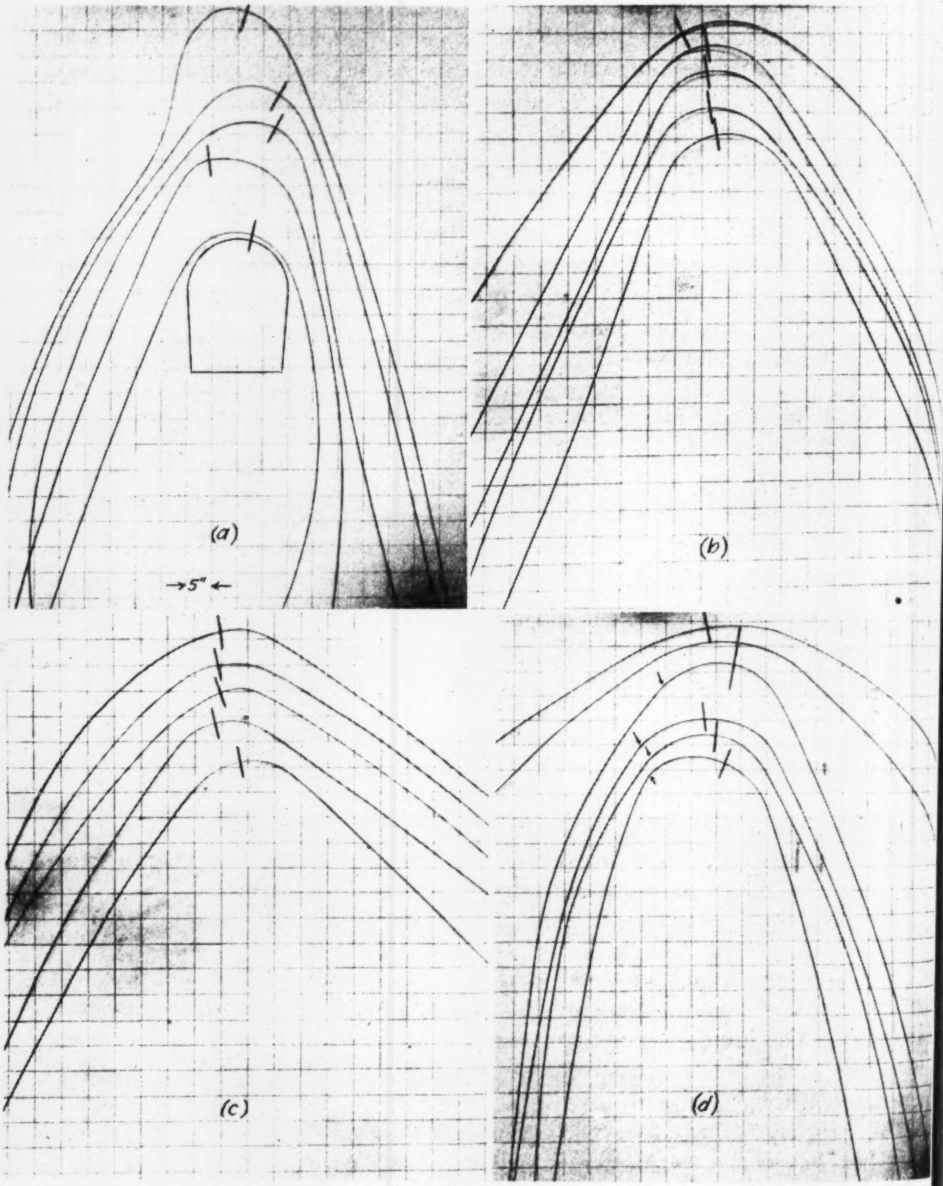
Of the several hundred specimens of the heat-treated wire broken in fatigue tests, the fact that in only one of them did the fatigue crack start from an internal nucleus is one item of evidence that internal cracks or fissures in the heat-treated wire were not a cause of its failure on the bridge. The large increase in fatigue strength obtained by machining away the outer layers is a further confirmation of this. If internal cracks existed in the heat-treated wire, they would be expected to act as nuclei for fatigue fractures to occur at approximately the same stresses at which the unmachined wire failed in the fatigue tests.

An intensive microscopical study of multiple longitudinal and transverse sections of numerous specimens of the heat-treated wire did not yield any positive evidence of the existence of internal cracks in the wire.

The results of the fatigue tests permit the conclusion to be made that if the fractures in the heat-treated wire at the anchor shoes on the bridge were fatigue fractures resulting from fluctuating tensile loads on the cable anchorages, they did not occur because the fatigue strength of the heat-treated wire was less than that of the cold-drawn wire which replaced it. There is no question but that the cold-drawn wire, which has proved to be satisfactory in the rebuilt cables, was subjected to the same loading conditions, during the reconstruction of the bridge, as were applied to the heat-treated wire when the bridge was being constructed originally. It does not follow, necessarily, that under the same loading conditions at the anchorages, the *stress* conditions in the portions looped around the anchor shoes were the same in the two types of wire.

STRESS CONDITIONS IN THE HEAT-TREATED WIRE AT THE ANCHOR SHOES

It was apparent that a knowledge of the stress conditions in the wire at the points where the fractures occurred, and during the time they were subject to the increasing load of the bridge, would be most useful to the investigation. Stress measurements on the wires had not been made at any time while they were on the anchor shoes. Nevertheless, specimens of the wire could be subjected, in the laboratory, to ranges of tensile load that bracketed the tensile loads on any of the wires at the anchor shoes. Likewise, the shapes of the loops of the wires around the anchor shoes, when under load, were known. Examination of the loops recovered from the bridge disclosed their shapes when unloaded. It was therefore possible to study the behavior of the wire when subjected to the deflections, from unloaded to loaded shape in the loops, caused by varying tensile loads.



(*a*) No. 3 strand, West Anchorage, Bristol.

(*b*) No. 2 strand, West Anchorage, Portsmouth.

(*c*) No. 3 strand, East Anchorage, Portsmouth.

(*d*) No. 3 strand, West Anchorage, Portsmouth.

FIG. 20.—Photographs of Terminal Loops of Heat-Treated Wire Removed from Anchor Shoes on Mt. Hope Bridge.

Pencil locates point on loop that was in contact with mid point on curved portion of shoe.

A record was made of the curvature of each of the unbroken heat-treated wires in 9 of the 28 strand loops recovered from the bridge, including the two with the greatest number of fractured wires and the two with the least number. The straight "legs" of each wire, extending beyond the anchor shoe, were cut off. The remaining curved portion was laid, without any lateral constraint, on a flat board on which were drawn rectangular grid lines. The point on the wire that had coincided with the mid-point of the circular bearing on the anchor shoe was marked. It was thus possible to determine the deviation between the curvature of the "strain-free" or unloaded wire and its curvature when snugged close to the anchor shoe. A total of 1181 wires were examined.

Photographs of four groups typical of the various degrees of deviation from the desired curvature are shown in Fig. 20. Not a single wire, of all those examined, was shaped accurately to the curvature of the anchor shoe. Many deviated but slightly from the originally straight wire; many had reverse curves; others contained rather sharp bends or kinks, as shown at the points indicated by arrows, Fig. 20 (*d*); very few had a regular curvature. In practically all of the wires, the point which had coincided with the mid-point of the circular curve on the anchor shoe was not at the mid-point of the curved portion of the wire. The preforming operation had failed completely in accomplishing its purpose of shaping the wire to fit the anchor shoe. In many cases the wire had been *deformed* so that, when snugged close to the shoe, bending stresses were induced that were greater than if no attempt had been made to shape the wire.

Failure to obtain a regular curvature could not be traced to irregular bending properties in the wire. Numerous 35-ft. lengths of the wire from the bridge were wound, under a constant tensile load, on a 9-in. diameter drum. The maximum difference in radius of curvature, from end to end of the wire, when released from the drum was less than $\frac{1}{2}$ in. Incidentally, it was found that the wire recovered from the bridge could not be shaped accurately to the curvature of the shoe by winding it on a $9\frac{1}{2}$-in. drum or sheave, but that by winding $1\frac{1}{4}$ turns on a sheave $8\frac{1}{2}$ in. in diameter the wire, when released, assumed a shape closely agreeing with the periphery of the anchor shoe. It is obvious, however, that failure to shape the wires properly, on the bridge, was not the result of using an incorrect size of preforming sheave.

The strand loops from the two anchor shoes where most of the fractures occurred, strand No. 1 of the east cable at the Bristol anchorage, and strand No. 3 of the west cable at the Portsmouth anchorage, each contained only about 40 unbroken wires. The average radius of curvature of these wires was distinctly greater than that in the wires of the remaining 7-strand loops examined. Sharp bends or "kinks" such as those shown in Fig. 20 (*d*) were found only in wires from these two strand loops. This is possibly significant in the explanation of the large number of fractures at these two anchor shoes.

The cold-drawn wire in the "hairpins" used to repair the broken wires on the bridge was curved regularly and uniformly on a 40-in. radius when released from lateral constraint. It is assumed that the cold-drawn wire at present in the cables had the same curvature when placed on the anchor shoes. It was evident that bending deflections in many of the heat-treated wires, when they were forced from the strain-free shapes shown in Fig. 20 to the shape of the anchor shoes by a tensile pull on the wire, were greater than the bending deflections imparted to the cold-drawn wires in the same manner. This was particularly the case for wires with practically no preforming, wires with reversed curves, and wires with sharp bends. It is highly significant that such irregularities causing the maximum deflections were located on those portions of the loops where most of the fractures occurred.

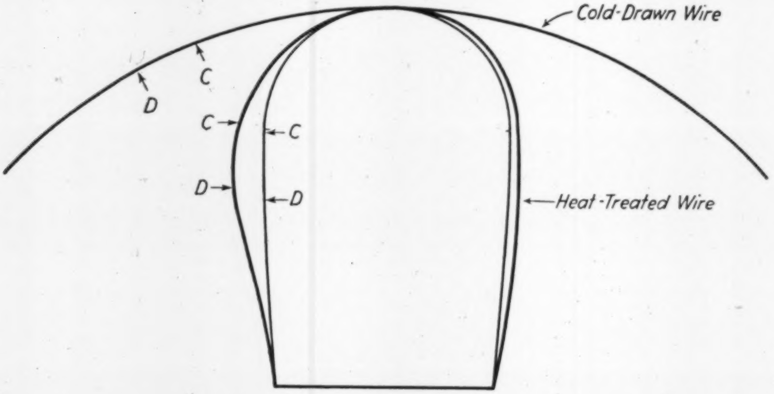


FIG. 21.—Curvature of Cold-Drawn Wire; of a Typical Terminal Loop of the Heat-Treated Wire; and of the Anchor Shoe, Drawn to Same Scale.

In Fig. 21 the curvature of one of the heat-treated wires recovered from a strand loop is shown, when free of lateral constraint. The curvature of one of the cold-drawn wires, used to replace a broken wire is also shown. The change in extreme fiber stress, in bending each of these wires to the curvature of the anchor shoe, was calculated for the length C to D by the following formula:

$$S = E\, r \left(\frac{1}{R_1} - \frac{1}{R_2}\right)$$

where S = maximum change in fiber stress in pounds per square inch,
E = modulus of elasticity (29,000,000) in pounds per square inch,
r = one-half diameter of wire in inches, and
$\left(\frac{1}{R_1} - \frac{1}{R_2}\right)$ = change in radius of curvature in inches.

The cold-drawn wire, between *C* and *D*, changed from a radius of curvature of 40 in. to one of 15 ft. The heat-treated wire, between *C* and *D*, changed from a radius of curvature of 11 in. to one of 15 ft. The corresponding changes in calculated extreme fiber stresses were 55,000 lb. per sq. in. for the cold-drawn wire and 242,000 lb. per sq. in. for the heat-treated wire. At the top of the loop, as shown in Fig. 21, the conditions were reversed, and a greater stress was produced in the cold-drawn than in the heat-treated wire. The curvature of the heat-treated wire shown in Fig 21 is typical of that of many of the unbroken wires in the strand loops recovered from the bridge. The calculated maximum fiber stresses, incurred in bending such wires to conform to the anchor shoes, were greater than the tensile strength. As the wires had not broken, it is evident that plastic deformation had taken place in the most severely stressed outer fibers, so that the incurred bending stresses were not those calculated from the formula, but were limited to the magnitude of the elastic range. The significant fact is that at the tangent points on the anchor shoes the heat-treated wires were subjected to greater deflections, and consequently to higher bending stresses than the cold-drawn wire.

TABLE XI.—MODULUS OF ELASTICITY AND PROPORTIONAL LIMIT (TENSION TESTS).

	Modulus of Elasticity, lb. per sq. in.		Proportional Limit, lb. per sq. in.	
	Heat-Treated Wire	Cold-Drawn Wire	Heat-Treated Wire	Cold-Drawn Wire
Average for 10 specimens	29 400 000	28 200 000	99 700	84 200
Maximum	30 100 000	28 800 000	111 500	105 200
Minimum	28 500 000	27 800 000	92 400	64 800
Average deviation from mean	±400 000	±300 000	±6 200	±8 000

A study of the stress-strain relations in tension tests of straight specimens of both types of wire also disclosed that for equal deflections (strain), the stresses were greater in the heat-treated than in the cold-drawn wire. Determinations were made on ten specimens of each type of wire. The loads were applied with a pendulum type hydraulic testing machine of 50,000-lb. capacity. Over the ranges used, it was found, by calibrations before and after the tests, that the indicated loads did not differ from the correct loads by more than +1 per cent. Extensions, over an 8-in. gage length, were measured with a Ewing extensometer. Calibration[19] showed that the extensions indicated by this instrument for equal increments of extensions produced by standard gage blocks did not differ by more than ±0.1 of one scale division, which was within the precision with which scale readings could be made. The specimens were loaded progressively, each increment of load being held only for a sufficient time to read the extensometer. The stresses were calculated from the cross-sectional areas of the wires with the zinc coatings removed. The proportional limits were determined by the method proposed by Tuckerman[20] using a deviation of 0.00002 in. per inch as a criterion. The modulus of elasticity of each specimen was computed as the average slope of the stress-strain curve to the proportional limit.

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[19] A. H. Stang and L. R. Sweetman, "An Extensometer Comparator," National Bureau of Standards *Journal of Research*, Vol. 15, No. 3, p. 199 (1935).

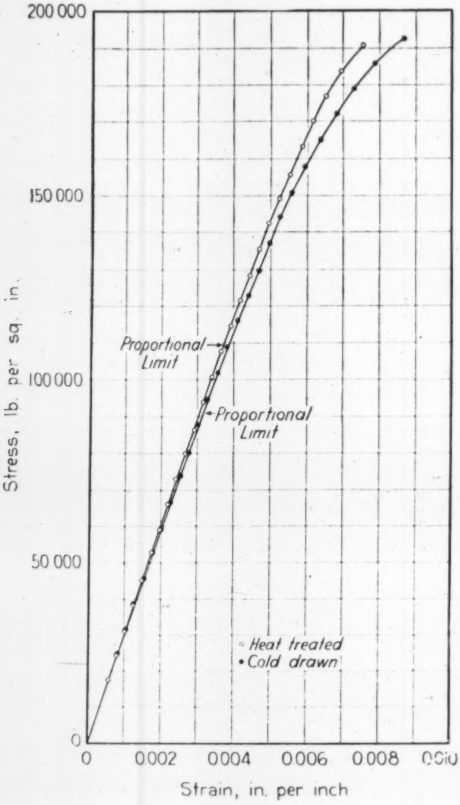

FIG. 22.—Stress-Strain Diagrams of Heat-Treated and Cold-Drawn Bridge Wire.

The results are summarized in Table XI. They show that both the modulus of elasticity and proportional limit were higher in the heat-treated than in the cold-drawn wire. A stress-strain curve for each type of wire is plotted in Fig. 22. The specimens from which the plotted data were obtained were those closest to the average for each group of ten specimens. Experimental determinations of the proportional limits did not determine the maximum tensile stresses that could be supported by the wires for an indefinitely long time without permanent set.

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[20] Discussion by L. B. Tuckerman of paper by R. L. Templin, "The Determination and Significance of the Proportional Limit in the Testing of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, pp. 538 (1929).

Determinations were made of the permanent set after release of tensile stresses maintained for 1-hr. and 48-hr. periods. An initial load of 500 lb. (16,000 lb. per sq. in. stress) was applied to the specimens to pull them straight. A new specimen was used for each determination. The load was increased gradually to the maximum, maintained constant for 1 hr., or 48 hr., and then decreased to 500 lb., when the permanent extension in the 8-in. gage length was measured with a Ewing extensometer. The results, given in Table XII, show that in the heat-treated wire there were no significant differences between the permanent extensions produced by a given stress maintained for 1 hr. and for 48 hr. In the cold-drawn wire the permanent extensions after 48 hr. at the higher stresses were appreciably greater than those produced in 1 hr. at the corresponding stresses. Of more significance is the fact that the permanent extensions in the cold-drawn wire were greater than those in the heat-treated wire. Creep at room temperature occurred in the cold-drawn wire at stresses below the yield strengths given in Table VI.

TABLE XII.—PERMANENT SET AFTER 1 HR. AND 48 HR. UNDER TENSILE STRESS.

Tensile Stress, lb. per sq. in.	Set in 8-in. Gage Length Stress Maintained 1 hr., in. per inch		Set in 8-in. Gage Length Stress Maintained 48 hr., in. per inch	
	Heat-Treated Wire	Cold-Drawn Wire	Heat-Treated Wire	Cold-Drawn Wire
33 000......	0.001	0.005	0.001	0.001
67 000......	0.003	0.008	0.003	0.008
83 000......	0.002	0.022	0.004	0.010
100 000......	0.004	0.026	0.013	0.018
117 000......	0.011	0.040	0.012	0.049
133 000......	0.021	0.056	0.016	0.061
150 000......	0.036	0.090	0.030	0.119
167 000......	0.090	0.186	0.073	0.255
184 000......	0.211	0.480	0.31	0.679

These results were confirmed qualitatively by observing the rates of change in radius of curvature of the two types of wire subjected to tensile loads while looped over circular supports similar to the circular portions of the anchor shoes on the bridge. The specimens of the heat-treated wire were initially straight; those of the cold-drawn wire were curved, initially, on a radius of approximately 40 in. It required a pull on the two legs of the loop equivalent to a tensile stress of about 35,000 lb. per sq. in. to bring the heat-treated wire into close conformity to the curved surface of the anchor shoe. This was true for straight specimens of the wire and for loops recovered from the anchor shoes on the bridge. Evidently because of the high elastic limit, these conditions were not altered by successive loading of the specimens, even after considerably higher loads had been applied. It required a pull of approximately the same magnitude on specimens of the cold-drawn wire, curved initially to a radius of 40 in., to bring the loop into close conformity to the curved support, but when the load was released after a short time, it could be brought back into conformity by a pull equivalent to only 20,000 lb. per sq. in. (approximately).

Loads producing tensile stresses in the wire of 50,000; 100,000; and 175,000 lb. per sq. in. were used in the experiments to hold the specimens to the curvature of the anchor shoes. At intervals over a period of 60 days the tensile loads were released and the successively smaller radii of curvature of the wires free of lateral constraint were measured. The strains incurred at the extreme fibers when the wires were deflected from their unconstrained shapes to the curvature of the anchor shoe were calculated from the formula:

$$E = \frac{d}{2}\left(\frac{1}{R_1} - \frac{1}{R_0}\right)$$

where E = strain at extreme outer fiber in inches per inch,
d = diameter of the wire in inches,
R_1 = radius of curvature of anchor shoe—$9\frac{3}{4}$ in., and
R_0 = radius of curvature of wire when free of constraint, in inches.

The results for both types of wire are plotted in Fig. 23. Each plotted point is the average value obtained from two or three specimens. Smooth curves were drawn to show the general trend of the changes in strain with time under load. It is evident that after the first ten days the strains incurred while the wires were constrained to the curvature of the anchor shoes were mainly elastic, and that the cold-drawn wire approached an equilibrium condition of no further appreciable change in radius of curvature earlier than did the heat-treated wire. It is likely that even the cold-drawn wire, in a very much longer time under load, would not have assumed the curvature of the support with no residual elastic strain. In the Final Report of the Board of Engineers to the Delaware River Bridge Joint Commission it was stated[21] that when the Niagara River suspension bridge was dismantled after about 50 yr. of service the cold-drawn wires of the cables tended to curve instead of lying straight. Residual elastic stresses are evidently present in all suspension bridge cable wires.

The experiments discussed above showed that the higher the applied tensile load the lower the unbalanced residual strain in both types of wire, but that in the heat-treated wire the strain was at all times greater than in the cold-drawn wire. Furthermore, the difference between the two types of wire was greatest at the lowest applied tensile stress. Although the specimens of heat-treated wire used in these experiments were initially straight, their curvature after 60 days under load was not significantly different from that of many of the unbroken loops recovered from the bridge.

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[21] Page 38 of the published report.

Studies of the stress conditions existing in those portions of the heat-treated wires looped around the anchor shoes on the bridge brought out the following facts:

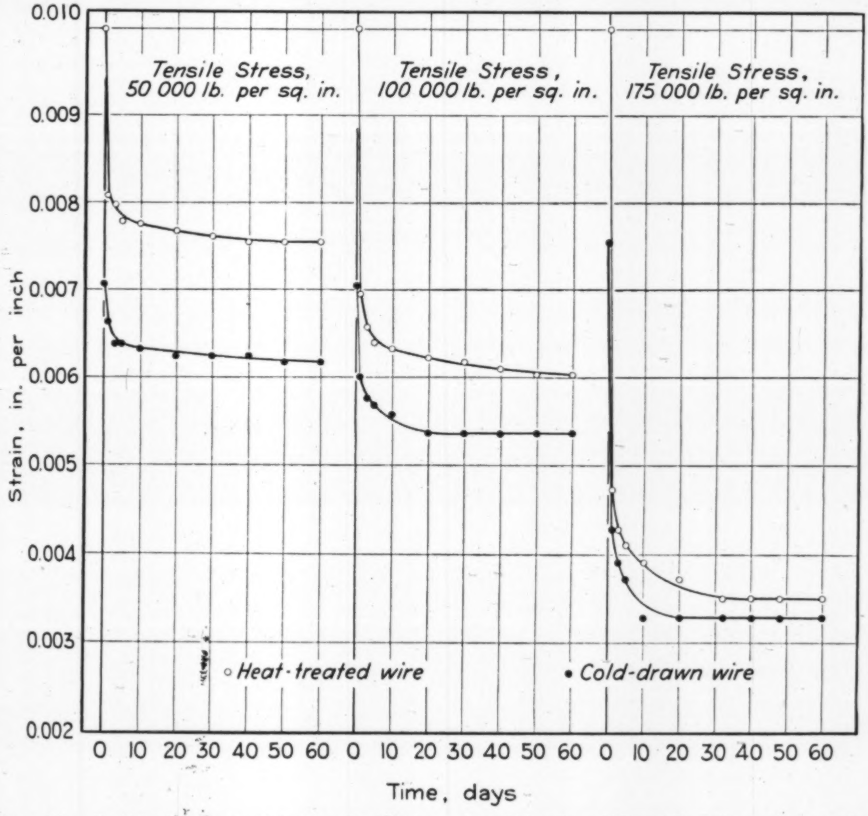

FIG. 23.—Residual Elastic Bending Deflections (Strain) when Wires Had Been Constrained to Curvature of Anchor Shoes for Various Times Under Tensile Loads on Loop Equivalent to 50,000; 100,000; and 175,000 lb. per sq. in.

1. Observations of the stress-strain relations in tension tests showed that for equal changes in radius of curvature the stresses were greater in the heat-treated than in the cold-drawn wire.

2. It was evident from the observations of the unbroken loops around the anchor shoes that the changes from the stress-free shape to the shape of the shoe were greater in many of the heat-treated wires than in the cold-drawn wire initially curved on a 40-in. radius.

3. Measurements of permanent set under loads sustained for 1-hr. and 48-hr. periods, and the behavior of the two types of wire when constrained to the curvature of the anchor shoes by tensile loads for 60-day periods showed that the stresses due to bending deflections were lower originally, decreased at a more rapid rate and to lower values in the cold-drawn than in the heat-treated wire.

4. These results were in accordance with the conclusion that the elastic bending stresses in the loops of the heat-treated wires around the anchor shoes were greater than those in the corresponding portions of the cold-drawn wires when the cables were replaced.

If the elastic bending stresses resulted from *constant* tensile loads, or from *continuously increased* tensile loads, applied by the pull of the cables on the anchor shoes, they were not sufficiently greater in the heat-treated wire to account for its failure on the same bridge where the cold-drawn wire has proved to be adequate. This was shown by the results of the tests described in the section on Long-Time Loading of Loops Supported on an Anchor Shoe.[22] It was found that elastic bending stresses equal to the maximum obtainable and acting in conjunction with tensile stresses resulting from loads applied at the ends of the loops did not cause failures in the heat-treated wire specimens at less than the nominal tensile strength of the wire. When the tensile stresses resulting from the progressively increased tensile loads became equal to the elastic limit, plastic extension took place, causing a decrease in the elastic bending stresses, and the tensile stresses then became the predominating factor in producing the fractures. The loop specimens reacted to this manner of loading in the same way as originally straight tension test specimens.

If, however, the tensile load on the wires exerted by the pull of the cables on the anchor shoes fluctuated from its value at any instant, the elastic bending stresses in the wires would also fluctuate over a range, provided that the change in tensile load permitted a change in the curvature of the wires where they were looped around the anchor shoes. The fact that it required a tensile stress of the order of 35,000 lb. per sq. in. on the legs of a loop of the heat-treated wire to bring it into close conformity to the curved portion of the anchor shoe indicates that the loops on the bridge had at no time conformed accurately to the anchor shoes around which they were placed. The tensile stress in the wire at the anchorages had been calculated as 32,000 lb. per sq. in. at the time the bridge was dismantled. Any change in the tensile load on the cable anchorages, either increase or decrease, caused a change in the curvature of the wires in the loops around the anchor shoes. The resulting change in fiber stress, because it involved a change in bending stresses, was greater than the change corresponding to the increase or decrease in tensile load. In such circumstances the magnitude of the range of stress would be the predominating factor in the event of failure of the wire. It would be immaterial whether the range of stress occurred in bending or in tension; if both types of stress were continuously within the elastic limit, their effect would be additive.

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[22] See p. 47.

It was known that the failures on the bridge had occurred without any plastic deformation at the fractures. It has been shown that the failures were not caused by the presence of gross defects in the wire. In the absence of gross defects, fractures without deformation in a ductile metal are obtained only when the metal has been subjected to repetitions of a range of stress exceeding the fatigue limit. The results of the tensile stress fatigue tests showed that the safe range of pulsating tensile stress was approximately 50,000 lb. per sq. in. It was entirely possible therefore that the safe range could be exceeded by fluctuations of bending and tensile stresses entirely within the elastic limit of the wire. A precise determination of the elastic limit, in tension, of the heat-treated wire was not experimentally practicable. The results of set measurements, Table XII, indicated that stresses up to 100,000 lb. per sq. in. did not produce significant permanent extensions in the heat-treated wire.

In the absence of actual measurement of stress in the wires at the anchor shoes during the construction of the bridge, only indirect evidence is available to show that during this period the tensile pull exerted on the anchor shoes by the cables did fluctuate. The following analysis of the available evidence is presented to establish this fact as a reasonable explanation of the mechanism of the failure of the heat-treated wire.

It is obvious that changes in live load and oscillations due to wind forces on a completed suspension bridge structure cause fluctuations in the tensile stresses in the cable wires at the anchorages. Daily and seasonal variations in temperature cause changes in length of the cables, resulting in fluctuations in tensile stresses at the anchorages, which are fixed. Fluctuations from such sources are of low frequency and are a small proportion of the total forces due to the dead load of the bridge structure.

During the construction period, before all of the stiffening trusses are in place, the different portions of the structure react elastically, independently of one another, and are free to oscillate through much greater amplitudes than is the case after all of the stiffening work has been placed, when the structure reacts elastically as a unit. Shifts in live load from one portion of the bridge to another, during construction, caused greater fluctuations in the tensile pull of the cables on the anchor shoes than was the case after the stiffening trusses were in place.

Probably the greatest oscillation due to wind forces occurred in the cables during the spinning operation and before any other portions of the bridge structure were suspended from them. It is a matter of record that the spinning of the original cables on the Mt. Hope bridge was interrupted on two days because of high wind, evidently because the cables were swaying, rather than because of a direct effect of wind velocity on the workmen. Visible oscillations of the cables were noted also by observers from the National Bureau of Standards at the time the bridge was dismantled.

These circumstances are believed adequate to establish the fact that oscillations of the cables in the spans caused fluctuations in the tensile pull of the cables on the anchor shoes, and therefore, as has already been pointed out, fluctuations in elastic bending stresses in the wires, around the anchor shoes.

It was evident that the maximum change in elastic bending stress would occur when there was a change in the tensile pull on wires that did not conform closely to the contour of the anchor shoe. Such conditions undoubtedly occurred during the construction of the bridge cables. If the dead load of the completed bridge had been added to that of the cables, slight decreases in the tensile pull on the anchor shoes, of the same order of magnitude as the decreases occurring in the early construction periods, might not have permitted elastic spring-back of the wires at the anchor shoes, and consequently no fluctuations in elastic bending stresses.

It was apparent therefore that if the wire was subjected to fluctuating tensile stresses, the most critical stage was during and immediately after the spinning of the cables, when the tensile loads were least. It was during this period that small changes in tensile load would cause the largest changes in elastic bending stresses at the anchor shoes.

The cold-drawn wire at present in the cables was subjected, during the reconstruction of the bridge, to tensile load fluctuations of the same type and magnitude that occurred in the original cables. But similarity in tensile pull of the cables did not result in similarity in the stress conditions in the two types of wire in the loops around the anchor shoes, for many of the heat-treated wires were subjected to greater changes in curvature, when constrained to the contour of the anchor shoes, than those necessary to make the cold-drawn wire conform to the same contour. It has been shown, also, that inherent differences in physical properties of the two types of wire caused the stresses produced by equal changes in curvature of the two types of wire to be less in the cold-drawn than in the heat-treated wire. Unfortunately, the lack of observations of the tensile loads on the wires at the anchor shoes during the construction period prevented a laboratory determination that the *range* of elastic bending stresses in the heat-treated wire, caused by fluctuations in the tensile loads occurring on the bridge, exceeded the fatigue limit, and that in the cold-drawn wire the *range* of elastic bending stresses was less than the fatigue limit.

It was possible, however, to determine the resistance of both types of wire to fluctuations in tensile loads that produced fluctuations of various magnitudes in the residual elastic bending stresses in the specimens. If fractures in the heat-treated wire similar to those that occurred on the bridge were produced by tensile load fluctuations of the order of magnitude that was possible during the construction period, the mechanism of failure of the wire on the bridge would be explained.

FLUCTUATING TENSILE LOAD TESTS ON LOOPS OF THE BRIDGE WIRE

Loop specimens of the wire were subjected to fluctuating tensile loads by alternately removing and replacing weights at the end of the lever arm *B* of the testing machine shown in Figs. 14 and 24. By the use of suitable counterweights various ranges of fluctuating loads were obtained at any desired mean load. A crank rotating at 20 to 25 r.p.m. raised and lowered the weight pan on the end of the beam. The specimens were looped over the semicircular support on top of the frame in the same way as for the long-time static loading tests described previously. Specimens of the heat-treated wire included unbroken terminal loops recovered from the bridge and also straight lengths from the original cables and from coils of new wire not used at the bridge site. The specimens which had not been preformed at the bridge were placed over the curved support, (*a*) without preforming; (*b*) preformed approximately to the curvatures of the terminal loops from the bridge (Fig. 20); or (*c*) preformed to fit closely to the curvature of the support. For the same conditions of curvature of the specimen and ranges of tensile load, no differences in results were observed between wire recovered from the bridge and the "new" wire. Specimens of the cold-drawn wire

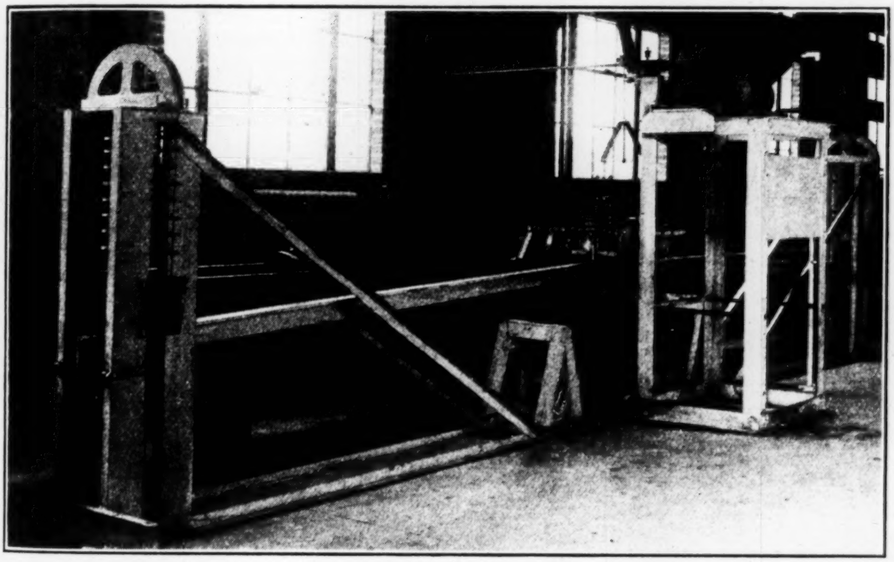


FIG. 24.—Photograph of Fluctuating Tensile Load Testing Machine.

were used, (*a*) with the original 40-in. radius of curvature; or (*b*) preformed to fit closely the curvature of the support.

A tensile stress of approximately 35,000 lb. per sq. in. on the unsupported "legs" of the loop was required to pull the specimens of both types of wire into position over the support so that a further increase in tensile stress caused no apparent change in curvature of the specimen. In the range 0 to 35,000 lb. per sq. in. small changes in tensile stress caused visible changes in curvature of the specimens. The greatest bending deflections occurred when the tensile stress was changed from 35,000 lb. per sq. in. or higher to the minimum obtainable in the apparatus. The specimens were not relieved of the weights of the yoke, grips, links, and a portion of the beam, which combined caused the minimum tensile stress obtainable to be approximately 600 lb. per sq. in.

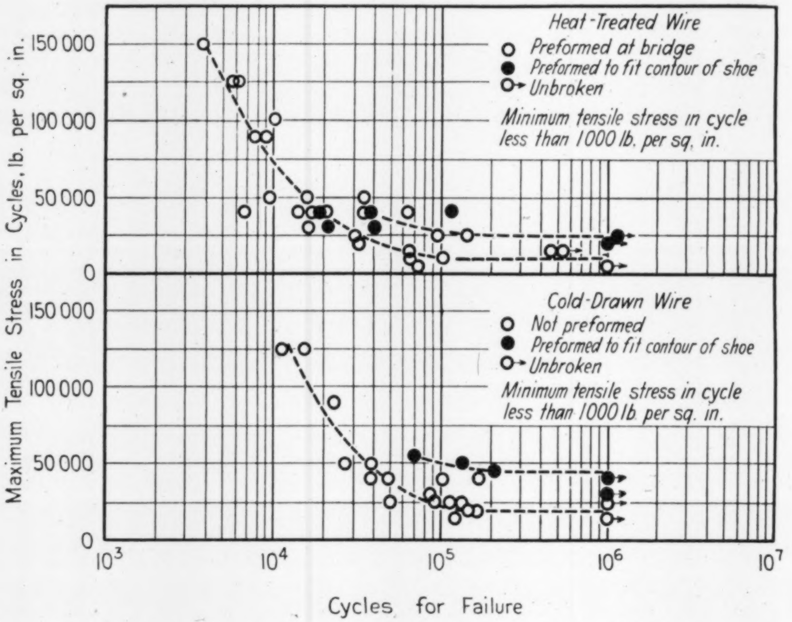
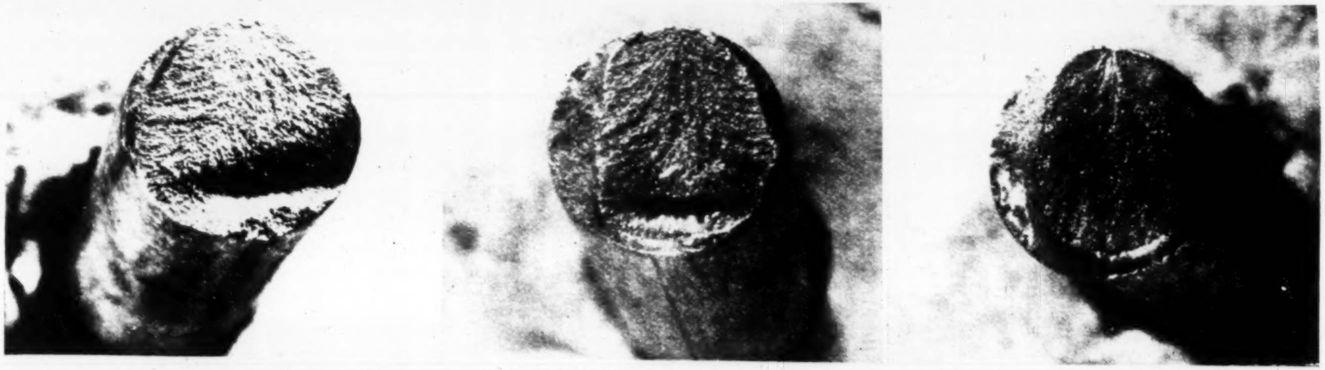
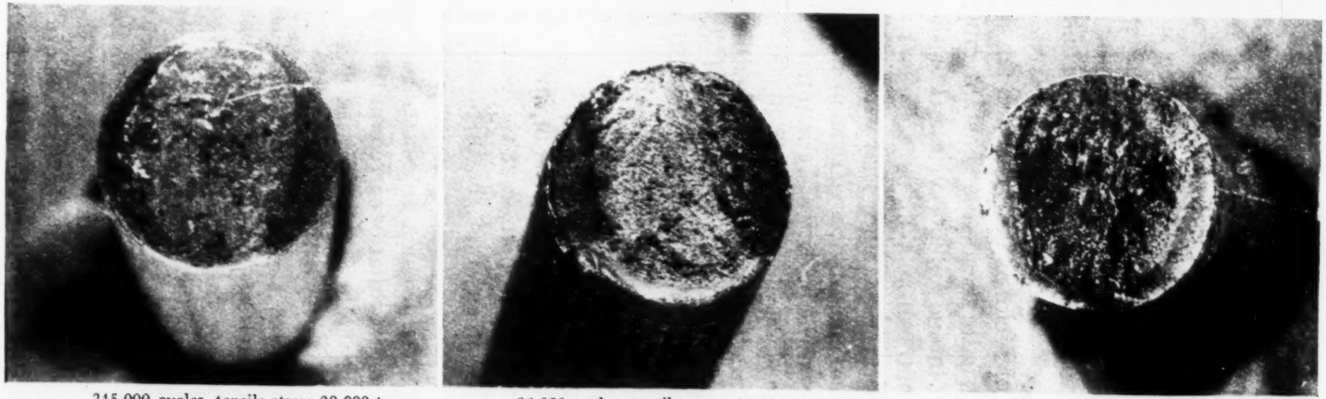


FIG. 25.—Stress-Cycle Graphs, for Ranges of Fluctuating Tensile Load 0 to Maximum on Legs of Hairpin.

Determinations were made of the number of cycles of various ranges of fluctuating tensile stress to cause fractures in the wire when the minimum tensile stress of the cycle was less than 1000 lb. per sq. in. (the least obtainable). The results for both types of wire are plotted in Fig. 25. In the graph for the heat-treated wire, the points on the lower curve were obtained with specimens taken from the strand loops around the anchor shoes, recovered from the bridge, and with specimens preformed in the laboratory to curvatures like those obtained from the bridge. The points on the lower



(*a*) Fractures that occurred on the bridge.



245,000 cycles, tensile stress 20,000 to 40,000 lb. per sq. in.

96,050 cycles, tensile stress 25,000 to 50,000 lb. per sq. in.

14,200 cycles, tensile stress 0 to 40,000 lb. per sq. in.

(*b*) Fractures obtained in laboratory.

FIG. 26.—Appearance of Fractures in Heat-Treated Wire That Occurred on the Bridge and Fractures in Heat-Treated Wire Obtained in Laboratory Under Fluctuating Tensile Loads (× 6).

curve in the graph for the cold-drawn wire were obtained with specimens that had a 40-in. radius of curvature when placed in the testing apparatus. The points for the upper curve of each graph were obtained with specimens that had been preformed in the laboratory to fit as closely as possible the curvature of the semicircular support and hang straight between support and grips. The galvanized coating was present on all specimens.

In general, at equal ranges of tensile stress, the cold-drawn wire endured a distinctly greater number of cycles before fracture than did the heat-treated wire. Although there is considerable scattering of the plotted points from a smooth curve, the "stress-cycle" graphs show the increase in number of cycles as the maximum tensile stress of the range was decreased, with a definite trend to a constant range within which fractures were not obtained in one million cycles. For the heat-treated wire, this range was only from 0 to 10,000 lb. per sq. in.

It is rather startling that wire with a tensile strength of 220,000 lb. per sq. in. and mounted as a tension member in the same manner as it was installed on the bridge, was fractured when the tensile stress on the straight legs of the loop did not exceed 10,000 lb. per sq. in. It was, of course, not the direct action of the range of tensile stress that caused the fracture, for it has been shown that the wire could be subjected safely to a range of pulsating tensile stress of approximately 50,000 lb. per sq. in. at mean stresses from 50,000 to 150,000 lb. per sq. in. The fractures were directly the result of the range of bending stress incurred when the tensile loads were alternately increased and decreased.

In all of the specimens of both types of wire, the fractures occurred without any deformation at the point of fracture. The fracture on many of the specimens had the distinctive characteristics of fatigue fractures with the point of origin at the surface of the wire. The appearance of others, three of which are shown in Fig. 26, was strikingly similar to that of the fractures that occurred on the bridge, three of which are also shown.

When the heat-treated wire specimens were preformed to fit the support, so that very little bending took place as the tensile loads were increased and decreased, the range of tensile stress permitting one million cycles without fracture was increased $2\frac{1}{2}$ times, as shown by the upper curve of the graph for the heat-treated wire, Fig. 25. Even under these circumstances, the range, 0 to 25,000 lb. per sq. in. was less than the safe range of pulsating tensile stress under purely axial loading conditions. This indicates that it was impracticable to preform this wire at the bridge so as to avoid damaging bending stresses in the loops at the anchor shoes, and a consequent decrease in the resistance of the wire to fluctuating tensile loads.

For the cold-drawn wire, not preformed, the range of tensile stress, when the minimum was less than 1000 lb. per sq. in., was also surprisingly low. When this wire was carefully preformed it was possible to raise the maximum tensile stress to 45,000 lb. per sq. in. without causing fractures in one million cycles. This range was almost as large as the safe range of pulsating tensile stress obtained in the Haigh machines on straight specimens.

When the minimum tensile stress of the cycle was maintained at the higher values, listed in Table XIII, the range also was greater, and the higher the minimum, the greater the range. This is readily understandable when it is considered that the greater the tensile load retained on the specimen, the less the bending deflection when the load was increased or decreased. At equal values of minimum tensile stress, the maximum stress of the range was consistently higher for the cold-drawn than for the heat-treated wire. Except for the results plotted in Fig. 25, the upper limits of the ranges given in Table XIII were based on runs of 500,000 cycles without failure of the specimen.

TABLE XIII.—RESULTS OF FLUCTUATING TENSILE LOAD TESTS ON HEAT-TREATED AND COLD-DRAWN WIRE.

Condition	Heat-Treated Wire			Cold-Drawn Wire			Number of Cycles Without Failure
	Minimum Tensile Stress, lb. per sq. in.	Maximum Tensile Stress, lb. per sq. in.	Range of Tensile Stress, lb. per sq. in.	Minimum Tensile Stress, lb. per sq. in.	Maximum Tensile Stress, lb. per sq. in.	Range of Tensile Stress, lb. per sq. in.	
Galvanized—preformed as at bridge...	less than 1 000	10 000	10 000	less than 1 000	20 000	20 000	1 000 000
Galvanized—preformed to fit shoe closely....	less than 1 000	25 000	25 000	less than 1 000	45 000	45 000	1 000 000
Galvanized—preformed as at bridge...	15 000	30 000	15 000	15 000	40 000	25 000	500 000
	20 000	40 000	20 000	20 000	45 000	25 000	500 000
	25 000	50 000	25 000	25 000	55 000	30 000	500 000
	30 000	55 000	25 000	30 000	70 000	40 000	500 000
	40 000	65 000	25 000	40 000	70 000	30 000	500 000
	50 000	80 000	30 000	50 000	80 000	30 000	500 000
	75 000	105 000	30 000	75 000	120 000	45 000	500 000
	100 000	130 000	30 000	100 000	130 000	30 000	500 000
Stripped—preformed as at bridge.....	15 000	45 000	30 000	15 000	45 000	30 000	500 000
	30 000	60 000	30 000	30 000	75 000	45 000	500 000
	100 000	135 000	35 000	100 000	135 000	35 000	500 000
Galvanized—braced to prevent bending....	less than 1 000	40 000	40 000	less than 1 000	45 000	45 000	500 000
	15 000	60 000	45 000	15 000	60 000	45 000	500 000
	30 000	70 000	40 000	30 000	75 000	45 000	500 000

A limited number of determinations was made on specimens of both types of wire with the zinc coatings removed by solution in acid. For equal values of minimum tensile stress, the range for the stripped wire of both types was greater than that of the galvanized wire. The increase, particularly at the lower minimum stresses, was greater for the heat-treated than for the cold-drawn wire.

It was evident that the tensile stresses alone could not have caused the fractures and that they were a direct result of bending stresses. Observations were made of the behavior of specimens subjected to fluctuating tensile loads, but so constrained that changes in curvature were eliminated or made negligible in effect. It has been shown that damaging bending stresses could not be avoided by preforming the specimens as accurately as practicable to the contour of the anchor shoe. Rigid braces fastened to both legs of the U-shaped specimens, as shown in Fig. 27, prevented flexing of the specimen as the tensile loads fluctuated.[23] Instead of breaking at from 500,000 to 1,000,000 cycles of tensile stress ranging from less than 1000 to 10,000 lb. per sq. in. it was possible to raise the maximum tensile stress of the cycle to 40,000 lb. per sq. in. without causing fractures in 500,000 cycles. At the higher minimum stresses shown in Table XIII the ranges permitting 500,000 cycles without fracture were of the same order of magnitude, and were almost as high as those obtained with straight specimens in the Haigh machines.

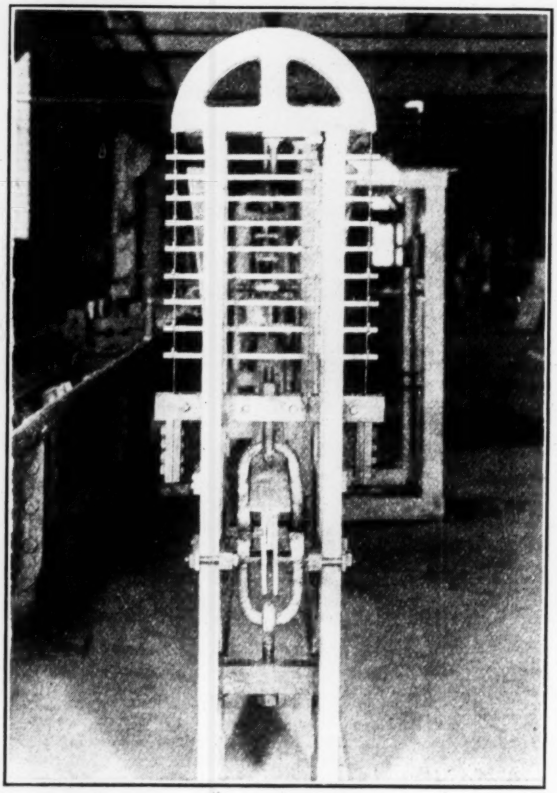


FIG. 27.—Braces on Legs of "Hairpin" to Prevent Bending of Wire as Tensile Loads Fluctuated.

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[23] The braces were steel bars ½ by 1 in. cross-section, drilled near both ends and slipped over the two legs of the "hairpin." The space between the wire and the inside wall of the holes in the bars was filled with Wood's metal, cast in place. In a few specimens, fractures were obtained at the point of contact between the Wood's metal and wire.

For cold-drawn wire, braced in the same way to prevent bending, the range of tensile stress permitting 500,000 cycles without fracture was not greatly increased over the range obtained at the same minimum tensile stress with specimens not braced. This is a further indication that in cold-drawn bridge wire, bending stresses which may be high initially, decrease more rapidly because of the lower elastic properties of the cold-drawn wire, than was the case with the heat-treated wire.

The results of the fluctuating tensile load tests showed that it was entirely possible to produce fractures in the bridge wire as the result of fluctuations in tensile load, when the tensile loads at no time exceeded the maximum calculated for the wire in the cables of the Mt. Hope bridge at the time it was dismantled. Furthermore, these experiments indicated that if the wires had been placed around the anchor shoes so that no bending stresses were incurred with changes in tensile load, practically the same resistance to fluctuating loads would have been obtained on the bridge that was shown possible with tests on straight specimens of the heat-treated wire.

The fractures on the bridge were the result of the same *kind* of stresses that produced the fractures in the fluctuating tensile load tests on the unbraced specimens in the laboratory. If it had been possible to apply higher initial tensile loads to the wire in the early stages of construction, the resistance of the heat-treated wire to fluctuating tensile loads would have been greater, possibly enough greater to have avoided the failures.

The laboratory tests showed that the resistance of cold-drawn bridge wire to fluctuating stresses was not greatly superior to that of heat-treated wire. The inherent ability of the cold-drawn wire to creep under stresses definitely within the elastic limit of the heat-treated wire indicates that the cold-drawn wire was not subjected continuously to elastic bending stresses of as high a range or magnitude as were present in the heat-treated wire when both were subjected to tensile loads of the same range and magnitude on the bridge.

Under the loading conditions imposed upon the terminal loops of the cable wires of the Mt. Hope bridge, the margin of safety of the cold-drawn wire was greatest in the early stages of construction, when the tensile loads were small.

DISCUSSION

Mr. A. V. de Forest.[1] This paper brings in the whole question of the elastic behavior of high elastic strength ratio steels under unusual fatigue conditions. The question is raised as to whether or not there is such a thing as static fatigue; whether you can have a slowly progressing crack under static loading which required days and weeks to show itself, and the relation of such cracks to improper preparation of surface.

There are at least two major lessons to be learned from this investigation. One is that hot galvanizing is a questionable method of preparing surfaces which are subject to fatigue or high static loads; and second, that locked-up stresses in highly elastic material must be more carefully controlled, and where this is not possible, a material of lower elastic *versus* tensile ratio is preferable.

Mr. E. E. Thum[2] (*presented in written form*).—In 1929, immediately after the report of failures on the Mt. Hope bridge, the present writer (then principal associate editor of *The Iron Age*) was able to devote his time uninterruptedly to an intensive study of bridge and spring wire. He concluded that the then discoverable facts fitted into the hypothesis that the fractures were due to an inherent characteristic of heat-treated, high-carbon steel wire (well-tempered martensite) after galvanizing—that is, that it is unable to withstand a slowly creeping load beyond the elastic limit. This hypothesis was discussed with numerous interested engineers and the investigators at the National Bureau of Standards, and formed the basis of several writings in *The Iron Age* and later in *Metal Progress*.

In suspension bridge cable, all straight wires curving around the anchorage shoes and some unfavorably placed ones squeezed under the cable bands at the suspenders are bent so that the extreme fibers are stressed beyond the elastic limit. This stress is never relieved—in fact, added to it is a slowly increasing tensile load as the floor system is completed. Similar conditions exist in cold-drawn bridge wire and the unsuitable heat-treated wire (although of somewhat less intensity in the cold-drawn variety, as Swanger and Wohlgemuth demonstrate), but the essential difference, in my opinion, is that the heat-treated wire is so stiff in its internal structure that it cannot yield readily at minor surface imperfections which exist in all galvanized wire at all parts of its length, and which act as "stress-raisers" at highly stressed elements such as mentioned just above. There is consequently no opportunity for plastic flow and redistribution of the stress to neighboring regions in the cross-section. Hence a splitting type of fracture with no contraction of area occurs under slowly creeping loads beyond the elastic limit. Cold-drawn wire, on the other hand, is much more plastic and has a low creep limit at atmospheric temperature, so the danger from scratches, notches or slag specks at the surface is much less.

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[2] Editor, ***Metal Progress***, Cleveland, Ohio.

If this hypothesis were true, it should be possible to duplicate the fractures occurring on the bridge by laboratory tests. H. F. Moore tried it at the University of Illinois on a few samples of galvanized spring wire—none of the suspicious bridge wire being procurable by him—with negative results (see *Metal Progress*, September, 1933). On the other hand W. A Slater and M. O. Fuller at Lehigh University tested nine pieces of the heat-treated bridge wire bent around a $9\frac{3}{4}$-in. radius and then loaded by increments starting at 84,000 lb. per sq. in. One broke while hanging overnight under the initial load, none withstood more than 201,500 lb. per sq. in., and all broke with a silky fracture, angular (no cup and cone), and no reduction of area.

As shown in Swanger and Wohlgemuth's paper, with step-up loads, starting at 90,000 lb. per sq. in., only eight broke at less than the nominal tensile strength but none less than 120,000 lb. per sq. in. (after an increment of 30,000 lb. per sq. in.), while 34 had less than 20 per cent reduction of area and therefore were classed by the investigators as a "brittle type of fracture." From their own tests Messrs. Swanger and Wohlgemuth concluded that "the results of the static [and step-up] loading under combined [flexural and tensile] stresses do not furnish an adequate explanation of the mechanism of failure of the wire on the bridge."

However, in the light of the tests at Lehigh and Washington, creeping loads cannot be *entirely* absolved from guilt. It is noteworthy that the difference between ultimate strength of heat-treated wire and its yield point is 33,200 lb. per sq. in., and the lowest breaks experienced at the Bureau of Standards occurred after stepping up the load 30,000 lb. per sq. in. Furthermore, the computed load on the cables at the Mt. Hope bridge when fractures were first found was 32,000 lb. per sq. in. This agreement of figures may be accidental, but then again it may have some significance as to the minimum tensile strength of this heat-treated wire when a tensile load is superposed on bending stresses beyond the elastic limit.

(It might be well to point out here that in the cable compacted within a suspender casting, or within the strand looped around an anchor shoe, or even in the bearing of a single wire against a semicircular casting, a considerable degree of external restraint is imposed. Whether heavy compression on the sides of a galvanized wire will have any effect on the tensile or endurance properties is unknown.)

But further consideration of the special conditions existing where the bridge wire starts bending around the anchor shoe (the locus of most of the trouble), Swanger and Wohlgemuth were led to the supposition that the tension was not relatively quiescent but rapidly fluctuating. Extensive fatigue tests on both heat-treated and cold-drawn wire were then made and developed their endurance as galvanized, as stripped, and as machined down to solid metal. It was discovered that a hairpin of heat-treated wire, bent around an anchor shoe and pulled taut, would break if the pull were fluctuated between 600 and 10,000 lb. per sq. in., whenever the experimental set-up was such that this range in tensile stress permitted the bent wire at the shoe to change its curvature by "spring back." In other words, minor changes in tensile stress induced major changes in flexural stress, beyond the endurance limit of the material, and fatigue failure resulted.

Since the investigators are able to break the heat-treated wire in laboratory tests at low tensile loads, and the fractures certainly look like many of those that occurred on the bridge, the presumption is very strong that they have hit upon the right track. The only questions left to consider are:

1. Did rapid fluctuations of 10,000 lb. per sq. in. actually occur in the bridge wire?

2. Why did so many wires break under the cable clamps at the suspender wires?

3. Why were the breaks at the anchorage concentrated largely in strand No. 1 of the east cable at the Bristol end of the Mt. Hope bridge, and in strand No. 3 at the Portsmouth end of the west cable?

In answer to question No. 1, it is unfortunate that Messrs. Swanger and Wohlgemuth could not extend their paper to include a mathematical analysis of the vibrations in the Mt. Hope bridge wire caused by side winds, the principal cause of rapid fluctuations, and an estimate of their effect on the tensile load. Lacking such analysis it is hard to imagine conditions that would cause a relief of 10,000 lb. per sq. in. in direct tension, when the stress in the wire due to its own weight is no more than 5360 lb. per sq. in. at the anchorage. Wind vibration effects would probably be worst during early stages of spinning, when the individual wires are hanging free, for as soon as a strand is spun it is bundled together at intervals along its length, and of course the complete cable is tightly compacted and bound into a quite stiff unit. Yet no breaks occurred in the cable in this early, flexible stage; they were first observed when the bridge was nearing completion and the whole thing was relatively rigid.

A corollary of the fatigue hypothesis is that unbroken hairpins recovered from the bridge should already be in a badly damaged condition and should break after a low number of additional stressings, as compared with hairpins made in the laboratory from straight wire. Perhaps the wide scatter of points in the S-N curve, top of Fig. 25, may be so interpreted, although Mr. Swanger has stated that he has found no consistent differences in test results ascribable to prior use on the bridge.

As to question No. 2, it is believed that the Bureau's investigators have paid too little attention to the breaks found along the cable, away from the anchorage. Since the total number of these breaks amounted to 31, they should not be lightly dismissed. One might well pause here to say—after the event—that *one* unexplained break is significant. I quote from *Metal Progress*, September, 1932: "During the development work on this wire, some 40-ft. lengths were hung over curved blocks on an idle crane girder and a platform on the lower ends loaded with billets, one or two added each day. Unexpected failures at the blocks and at low loads and deficient ductility resulted." It is not too much to believe that if these "unexpected failures" had been *properly* explained, the heat-treated wire would never have been shipped to the bridge site.

Conditions were somewhat different at the Detroit-Windsor bridge made of the same wire. *Engineering News-Record*, October 10, 1929, says: "Numerous breaks were subsequently discovered under the cable bands in the main strand at Detroit. . . . The principal difficulty in dismantling the main strand was occasioned by broken wires found at the suspender points; these wires became loose and entangled and others in one strand were so tightly squeezed to another strand that they had to be burned separately."

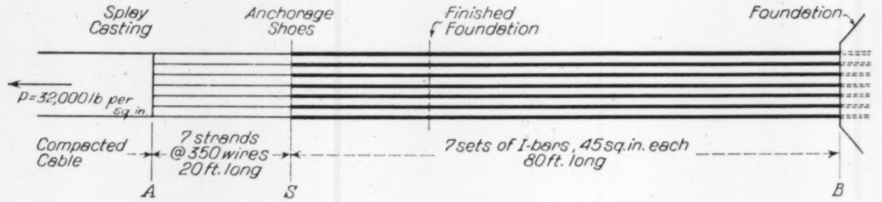
The creeping stress theory was adequate to explain these breaks in rather sharply curved wires near the surface, where the cable is squeezed tighter by the bands, and the slowly rising stresses of subsequent bridge erection. If fluctuating tension in bent wires is responsible for the breaks (as Messrs. Swanger and Wohlgemuth infer), the action cannot commence until the wires actually *are* bent (that is, until the clamps are tightened up), and consequently the damage started much later than at the anchorages and fatigue failure occurred in a comparatively short time in stiff, compacted cable—all of which seems rather unlikely.

The third question concerns the concentration of so many breaks in one strand of a cable. It is yet to be explained. However, a little elementary mechanics and algebra will indicate significant changes in the unit load in the unbroken wires in a badly damaged strand.

A well-compacted cable is a unit. For instance, it is almost impossible to pull out without breaking individual wires whose ends protruded from a 10-ft. length of cable made up for test purposes. The frictional resistance, wire to wire, is so great that for preliminary computation one can treat a compacted cable as a unit. Suppose that such a compacted cable is made up of seven rods and that each rod is properly anchored to an unyielding foundation. If one of these rods breaks at one end, the total load at that end is transferred to the six remaining rods. That is to say, the unit load increases on the wires at an anchorage where there are broken ones, but the stress conditions at the undamaged end of the cable are not influenced by changes at the other end, short of entire collapse of the bridge. It follows, therefore, that a bundle of wires such as strand No. 3 in the Mt. Hope bridge can be three-quarters broken at the Portsmouth end without requiring anything to happen at the Bristol end.

At the Detroit bridge the pit enclosing the eye bars and foundation girders had been filled with concrete up to the first pin. The elastic section then consisted of about 21 ft. of eye bar, concrete to shoe, and 30 ft. of wire, shoe to splay casting. Only a few broken wires were found at the anchorages on this bridge. At the Mt. Hope bridge each of the strands was anchored to two 12 × $1\frac{7}{8}$-in. eye bars, 80 ft. long. They were to have been concreted in for 60 ft. of this length, but in describing the installation of hold-backs prior to dismantling the bridge, *Engineering News-Record*, October 10, 1929, says, "Fortunately these eye bars had not been concreted in." The subsequent analysis will show whether this was "fortunate" or not; it also may give some clue as to why there was no undue concentrations of breaks in individual strands on the Detroit bridge.

General conditions therefore are as sketched below:



Prior to the time when any trouble occurred, the loading on each of these seven units was approximately uniform, and the elastic extension of each unit the same. Under these conditions the entire cable is carrying 2,368,000 lb. tension, each strand and mating eye bars 337,300 lb., each wire 962.9 lb., and each loop 1925 lb.

It is now required to find out what redistribution of stress will occur when a number of wires break in a single strand. Assume conditions in the east cable, Bristol anchorage, when 20 out of the 175 loops in strand No. 1 had broken, the whole cable being loaded at 32,000 lb. per sq. in. Instead of 2450 wires carrying the load there are now 2410, one broken loop putting two wires out of commission (both legs of the hairpin).

If the load on each wire in the six undamaged strands is M and the load on each wire in the damaged strand is N, then

$$2100\ M + 310\ N = 32{,}000 \text{ lb. per sq. in.} \times 74 \text{ sq. in.}$$

Also the load on each set of eye bars for each undamaged strand is 350 M and the set holding back the damaged strand is 310 N.

We can write an expression for the total elongation in the undamaged strand and its set of eye bars under the above loading, and this must equal the elongation in the damaged strand and its eye bars, since we have postulated no slippage in the compacted cable and no yielding in the foundation. This gives the second equation:

$$\frac{M \times 20 \times 12}{0.03 \times 29{,}400{,}000} + \frac{350M \times 80 \times 12}{45 \times 29{,}400{,}000} = \frac{N \times 20 \times 12}{0.03 \times 29{,}400{,}000} + \frac{310N \times 80 \times 12}{45 \times 29{,}400{,}000}$$

Solving this simultaneously with the first equation given we have:

M = 975.2 lb. equal to a stress of 32,400 lb. per sq. in.
N = 1032.2 lb. equal to a stress of 34,300 lb. per sq. in.

In other words, fracture of 20 loops has increased the load on the wires in the undamaged strands only 1.5 per cent, but due to the elastic contraction of the large eye bars, the remaining wires in the damaged strand had their load jumped up 7.2 per cent.

Conditions were much worse on February 22, 1929, the first day when a careful search was made, and 65 broken wires were discovered in strand No. 1 of the east cable at the Bristol anchorage. Using the above method of computation, the wires in the practically intact strands were stressed to 33,100 lb. per sq. in., (an increase of 2.9 per cent over a completely sound bridge) but the remaining sound wires in the failing strand carried 40,400 lb. per sq. in., an increase of 26 per cent.

It is a matter of record that on the very next day "three-quarters of the wires were broken in that particular strand," and haste was made to lighten the stresses by hold-back clamps and taking all movables off the bridge. Taking the final conditions as found in the east cable of the Bristol anchorage (140 broken wires in strand No. 1, the rest practically undamaged), the stresses were: In wires in undamaged strands 35,400 lb. per sq. in., an increase of 10.6 per cent; in wires in strand No. 1, 57,800 lb. per sq. in., an increase of 84 per cent.

At the Portsmouth anchorage, ten breaks were found in strand No. 3 on February 27, and the number increased to 129 on March 9, despite efforts to lighten the loads on the wire by bringing in shore all movables, from the structure.

Evidence from the Detroit bridge and both anchorages of the Mt. Hope bridge indicates that the concentration of breaks in definite strands occurred rapidly, and were associated with a rapidly increasing tensile load on the remaining sound wires in the damaged strand. A reasonable conclusion, therefore, is that fatigue fractures occurring more or less at random among the various wires happened to congregate in certain strands at two anchorages at purely accidental location. Once a considerable number were so concentrated (say 20) the whole strand was doomed, not because of fatigue fractures, but because of elastic contraction of the long eye bars and the induced creeping tensile stress in the remaining sound wires.

This rather brief review of some pertinent facts in this historic bridge failure indicates that, despite the enormous amount of careful and well-planned work done at the Bureau of Standards, a completely satisfactory explanation of the difficulty is not yet forthcoming. As before mentioned, the ability to develop fractures in the laboratory that look like the broken wire-ends from the bridge anchorages and at a combination of high bending and low tension loads indicates that we are close to the correct solution. In a number of instances, other factors also had major influence. What these factors are is still hypothetical. One may be heavy lateral constraint against movement at the surface. Another probably is a slowly creeping tensile stress.

Vulnerable though the bent heat-treated wire has proven to be to fluctuating load with unchanged average intensity, it may be even more vulnerable when the average intensity is gradually rising. A series of "step-up fatigue tests" would determine this point.

Lastly, the surface condition of the heat-treated wire was shocking. This refers not to the minor pits and scratches but to the deeper cracks which infested the wire and caused one out of every seven to ten tensile specimens to break with practically no reduction of area. Even though Messrs. Swanger and Wohlgemuth cannot find that these cracks affect the endurance of the wire more than the minor imperfections present everywhere, the deep cracks must have been responsible for a large proportion of the rejects at the mill, and this would have been more ruinous if retests had not been allowed. (Seventeen per cent of this wire was rejected, as compared with 1.5 per cent rejects of cold-drawn wire shipped from the same mill to the Delaware River bridge!)

Spring makers find similar cracks in the surface of heat-treated springs after electroplating—possibly it is a characteristic action of the cleaning acid on the surface of any heat-treated wire, no matter how well made. However the prevalence of such cracks as shown in Figs. 16 and 17 is possibly sufficient to place one of these major surface defects within the region of stress concentration for many of the breaks that actually occurred on the bridge, and possibly was the first cause of the entire trouble.

Now what metallurgists and designers would like to know, even though bridge engineers will wisely stick to cold-drawn wire, is whether heat-treated wire with really good surface, either bare or rust proofed without surface damage, would be immune to the troubles that plagued the Mt. Hope bridge? In other words, were the failures due to the essential nature of the microstructure (relatively stiff and unyielding) or is this characteristic harmless unless the metal has gross surface imperfections?

Even though some questions remain unanswered, the researches so far have clear lessons that point to certain desirable changes in the design of large suspension bridges. The relatively small margin of safety of cold-drawn wire (accepting all the implications of the endurance diagram, Fig. 25) indicates that both bending and fluctuating stresses should be minimized as much as possible. This will involve three things:

1. Anchor shoes should have as large a radius as possible.

2. Stiff "keepers", carefully machined to correct line and curvature, should enclose all strands from within the splay casting to and around each anchor shoe.

3. Eye bars should be concreted into the foundation as far up toward the anchor shoe as practicable.

Lastly, the practice of allowing a retest should be closely scrutinized. A plausible argument might be made for a retest when the original one fails to meet the specified values by a small margin once in a hundred times. But nothing can be said of a practice which permits the acceptance of material which shows deficient ductility in 10 per cent of the tension tests.

MR. H. F. MOORE.[3]—The details of the first series of long-continued static load tests on heat-treated spring wire, to which Mr. Thum refers as having been made at the University of Illinois, were published in the September, 1933, issue of *Metal Progress*. Since those tests, further tests were made on specimens of this same heat-treated spring wire after it had been hot-galvanized at the Battelle Memorial Institute. We were unable at that time to get samples of either the Mt. Hope or the Ambassador bridge wires, and studied a wire of about the same chemical composition heat treated to about the same strength as that reported for the bridge wires. As Mr. Thum reports, we were unable to find any evidence of any spreading crack under steady load, although preformed specimens and non-preformed specimens were both tested in the atmosphere and also under streams of fresh and salt water.

MR. B. L. MCCARTHY.[4]—I am inclined to concur with Mr. Thum in his views regarding the importance of low load stresses and their effect on certain areas. In our work we do a lot of oil tempering and the fact that we have encountered the same type of fractures as occurred in the bridge wire prompted these remarks.

We have found that the size of the austenitic grains developed in the tempering treatment is important and that low stresses tend to promote this type of fracture when the austenitic grain size produced in the hardening furnace is too large. This shows up sometimes in fractures resulting from strains set up in coiling the wire. These fractures sometimes occur overnight, sometimes it takes weeks to develop. The correction is in the proper heat treatment.

Another feature which has an important bearing on the austenitic grain size developed in hardening is the amount of strain distribution in the wire prior to tempering. The drafting from 0.233 to 0.192 in. is too small to produce uniform strain distribution. We have found that a non-uniform strain distribution in the wire will promote non-uniform grain coarsening when heat treated. This may produce abnormally coarse austenitic grains.

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[3] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.
[4] Metallurgist, Wickwire Spencer Steel Co., Buffalo, N. Y.

It is my thought that combining a small amount of cold work with a coarse grain or medium coarse grain steel as determined by the McQuaid-Ehn grain-size test would result in a grain-coarsening temperature below 1550 F. and produce the non-uniform grain size.

The low stresses seem to be effective along the old austenitic grain boundaries in oil-tempered wire, while the stresses set up in drawing cold-drawn wire are sufficient to stress the whole cross-section.

This results in a more uniform distribution of the stresses in the cold-drawn wire as compared with the oil-tempered wire. The ordinary tests such as fatigue and tension tests, set up stresses beyond the low stresses encountered in bending or in bridge wire and are effective throughout the cross-section and not along the old austenitic grain boundaries. This, in my opinion, accounts for the fact that physical tests did not reveal the true condition of the wire when low load stresses were employed.

MR. M. GENSAMER.[5]—In answer to Mr. McCarthy, in connection with austenitic grain size, we had a double heat treatment to take care of that. The first lead pan was just a pre-heat pan. The second lead pan took the wire above the critical temperature. The third lead pan lowered the temperature of the wire to below the critical temperature, and the fourth one brought it again above the critical temperature for the purpose of quenching the wire. I think this double heat treatment eliminated the possibility of any excessive grain size as a result of the small amount of cold work which the wire was given prior to the heat treatment.

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[5] Metals Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa. Formerly Metallurgist, Page Steel and Wire Co., Monessen, Pa.

HIGH-VELOCITY TENSION-IMPACT TESTS[1]

By H. C. Mann[2]

Synopsis

Data are presented which definitely show that high-velocity tests are essential to reveal the true dynamic properties of materials. The results obtained further indicate that in the selection of materials for use under conditions involving dynamic loading the following three significant factors must be considered:

1. The transition velocity within which normal material behavior may be expected.
2. The ratio of total energy value to mass or volume of material involved.
3. The effect of surface irregularities such as tool marks, size and form of fillets, etc.

The high-velocity tension-impact machine designed and built for this investigation is described, and the mathematical derivation of the equation used to calculate the energy required for rupture is given.

Diagrams and photographs of typical fracture transitions are presented, showing the effect of velocity on both ferrous and non-ferrous materials.

The impact test has always been a subject of controversy and disagreement among engineers and metallurgists. Although normally used as an acceptance limit, the results have seldom been found to be a definite guarantee of material serviceability under all conditions.

The effect of velocity on the energy value obtained from the impact test has usually been considered of minor consequence, and practically entirely disregarded in the past. While it had been suggested from time to time that tests at high velocity might develop valuable information, the results of an elaborate series of tests by Charpy and Cornu-Thenard,[3] carried out at velocities of from 2.5 to 21.5 ft. per sec., showed no appreciable energy differences, which was considered definitely to settle the question and discount the velocity factor as being of any great importance.

One fact, however, which appears to have received no consideration whatosever, was that no correlation had been discovered between the static and dynamic tests, and since the ordinary types of impact machines operate at only one velocity, there could be no way for determining whether the energy value obtained was equal to or less than the maximum resistance the material was capable of offering.

From the results of a series of investigations carried out at the Water-

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[1] Publication approved by the Chief of Ordnance, U. S. Army.

[2] Senior Materials Engineer, Watertown Arsenal, Watertown, Mass.

[3] Charpy and Cornu-Thenard, "New Experiments on Shock Tests and on the Determination of Resilience," *Journal*, Iron and Steel Inst., Vol. XCVI, No. 11 (1917).

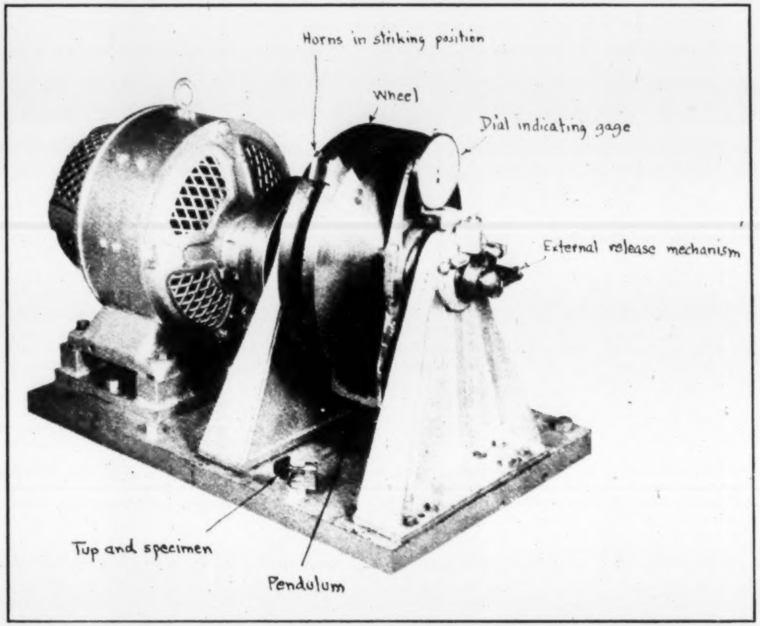
Horns in striking position
Wheel
Dial indicating gage
External release mechanism
Tup and specimen
Pendulum

FIG. 1.—High-Velocity Tension-Impact Machine.

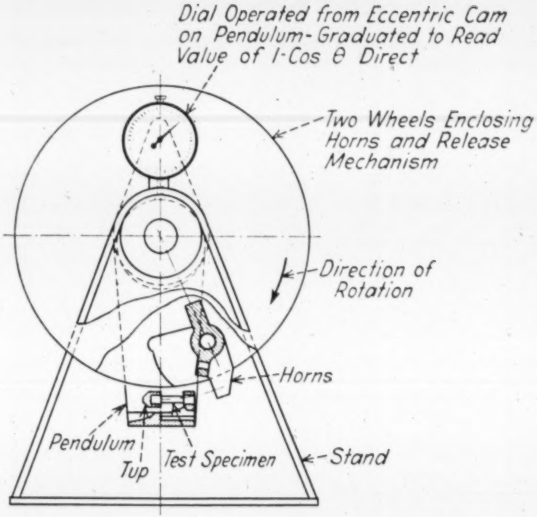
Dial Operated from Eccentric Cam on Pendulum- Graduated to Read Value of 1-Cos θ Direct
Two Wheels Enclosing Horns and Release Mechanism
Direction of Rotation
Horns
Stand
Pendulum
Tup
Test Specimen

FIG. 2.—High-Velocity Tension-Impact Machine.

town Arsenal Laboratory, a correlation between these two types of tests was discovered[4] which provided this necessary basis for comparison. This work also brought out the fact that velocity is an important factor which must be considered if the true dynamic properties of materials are to be determined.

In these tests, however, the maximum velocity available was only 28.5 ft. per sec., which was found to be insufficient to definitely reveal the velocity effect in any but a few materials. A special machine capable of producing velocities up to 1000 ft. per sec. was therefore designed and built for this purpose.[5] A general view of this machine and a sectional diagram indicating the operating details are shown in Figs. 1 and 2.

DESCRIPTION OF MACHINE

In principle this machine consists of two entirely separate and distinct parts: the pendulum as a means for measuring the energy required to rupture the test specimen, and the rotatable wheel and related parts which serve to develop the necessary kinetic energy and velocity. These two systems have no interconnection or effect on each other except through the test specimen during the process of rupture.

The pendulum which acts as the energy measuring system, is normally at rest, and moves only as a result of the impulse imparted to it through the test specimen, which is attached to and acts only on the pendulum. All other parts of the machine including the wheel, horns, tup, etc., act merely to develop and transmit energy to the free end of the specimen.

The wheel consists of two disks mounted on a hollow shaft, and separated by a sufficient amount to enclose retractible horns and balancing counterweight positioned diametrically opposite, and which revolve outward on pins attached to the disks. The horns and counterweight are held in the retractible position by a cross shaft fitted with interrupted threads, and controlled through the hollow shaft by an external tripping device, which at any desired velocity operates to release the horns and counterweight, permitting them to revolve outward due to centrifugal force.

In operation, the test specimens and tup are attached to the pendulum, and with the horns and counterweight retracted, the wheel is brought up to speed by means of a motor geared to the wheel supporting shaft. At the desired velocity, measured by means of a tachometer, the external mechanism is tripped, releasing the horns which revolve outward due to centrifugal force, striking the tup and thereby rupturing the test specimen. The angular movement produced in the pendulum is recorded on a special dial indicating gage, actuated by the rise of an eccentric cam attached to one side of the pendulum, this gage being graduated to read directly the value of $(1 - \cos\theta)$.

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[4] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

[5] R. K. Haskell and H. C. Mann, Variable Speed Tension Impact Machine, U. S. Patent 2,022,666, December 3, 1935.

The energy required to rupture the test specimen is determined from the following equation, derived from a mathematical calculation involving the constants of the machine, the angular velocity of the wheel, and the angular movement of the pendulum:

$$E = \omega_0(1 - \cos\theta)^{\frac{1}{2}} K_1 - (1 - \cos\theta) K_2$$

where E = energy required for rupture,
K_1 and K_2 = constants of the machine,
ω_0 = angular velocity of wheel, and
θ = angle of pendulum swing

The derivation of this equation is shown in the Appendix.[6]

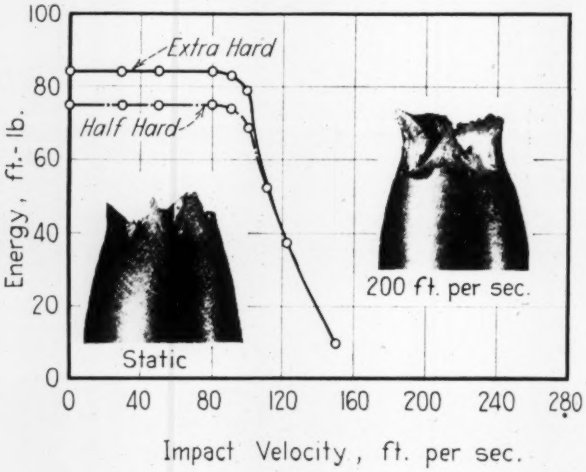

FIG. 3.—High-Velocity Tension Impact, Rolled Silicon Bronze.
Specimens, 0.252 in. by 1.0 in. gage.

TEST RESULTS

Except as noted in the figures, all specimens tested were $\frac{3}{4}$ in. round by 3 in. long, the ends threaded, and the middle section reduced to 0.252 in. diameter for a length of 1.0 in. In this form of specimen the end sections are sufficiently large to confine the work of deformation to the reduced section.

The results obtained from tests of rolled silicon bronze in the extra hard and half hard tempers are shown in Fig. 3. It will be noted that the energy values are constant up to a velocity of about 90 ft. per sec., and then suddenly decrease until at 110 ft. per sec. they have both dropped to 55 ft-lb., and at 150 ft. per sec., to 10 ft-lb. When tested at a velocity of 200 ft. per sec. there was no appreciable movement of the pendulum, indicating an extremely small amount of energy was required for rupture. From an examination of the fractured specimens it was observed that, within the

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[6] See p. 95.

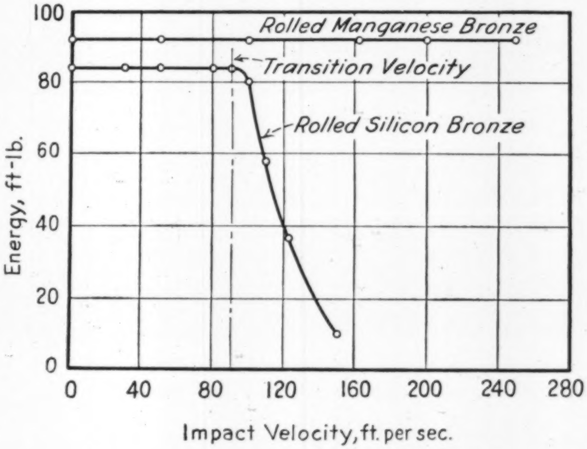

FIG. 4.—High-Velocity Tension-Impact Tests.
Specimens, 0.252 in. by 1.0 in. gage.

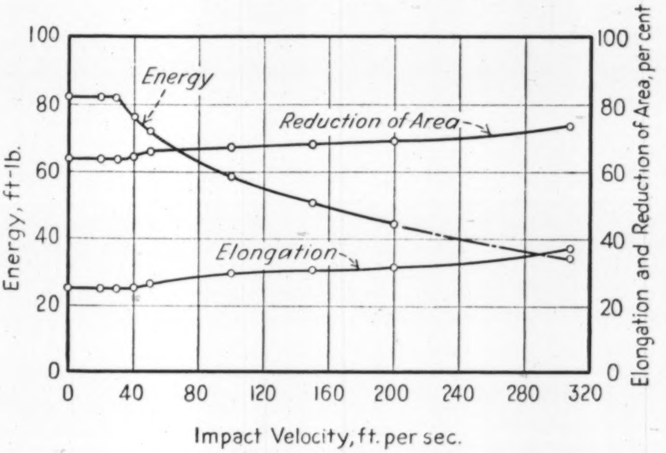

FIG. 5.—High-Velocity Tension Impact.
Specimen, 0.252 by 1.0 gage.

Chemical Analysis, per cent:

Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium
0.37	0.53	0.016	0.021	0.170	0.07	0.10

Heat Treatment:
1500 F., 1 hr., furnace cool to 1000 F., then air cool.
1280 F., 2 hr., furnace cool to 1000 F., then air cool.

transition velocity of 90 ft. per sec., the type of fracture was irregular along Lüder line formations. At higher velocities, however, the break assumed more and more the cupped type, and at 200 ft. per sec. it showed a deep cup and cone with complete absence of the Lüder lines.

A comparison with rolled manganese bronze is shown in Fig. 4. It will be noted that, while silicon bronze shows a transition velocity at 90 ft. per sec., the manganese bronze is unaffected up to a velocity of 250 ft. per sec. Since tests at higher velocities have not as yet been carried out on this material, its transition point is at present unknown.

The results of tests of S.A.E. No. 1035 steel in the annealed condition are shown in Fig. 5. In this case the transition velocity occurs at 30 ft. per sec. The significant fact brought out by the tests of this material is that above the transition velocity, as the energy values decrease, the ductility, as measured by elongation and reduction of area, steadily increases.

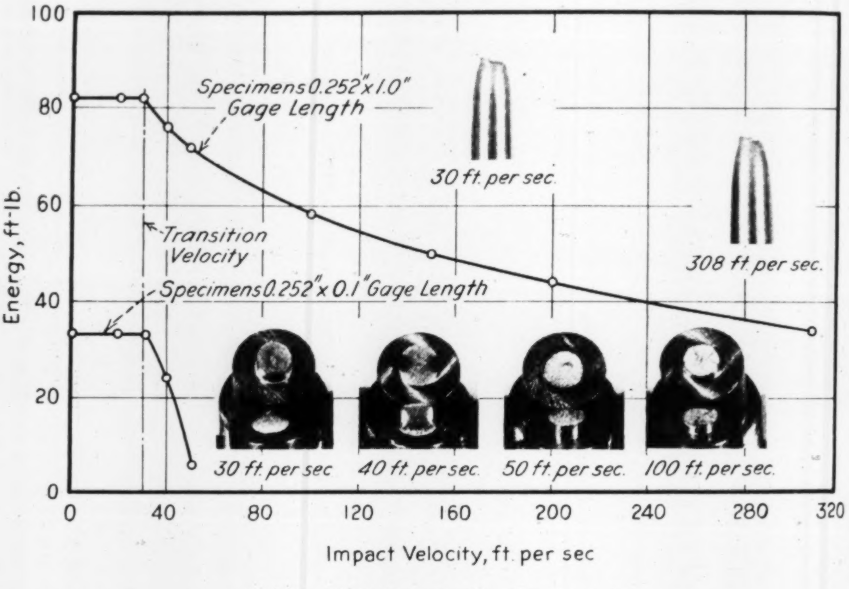

FIG. 6.—High-Velocity Tension Impact.
S.A.E. No. 1035 Steel, Annealed

To determine the effect of specimen form on the transition velocity, the gage length was shortened to 0.1 in., giving the form of a square notch. A comparison with the results from the 1.0-in. specimens is shown in Fig. 6. It will be noted that the transition velocity is the same in both cases, which appears to correlate this factor as a function of material condition. The short gage length specimens, however, exhibited less ductility at velocities above the transition point, the fracture changing from a perfect cup at 30 ft. per sec. to a 45-deg. shear at 40 ft. per sec., at 50 ft. per sec. to a coarse crystalline fracture at the corner, and at 100 ft. per sec. to a fine crystalline break directly at the middle of the 0.1-in. gage length.

To investigate further the probable effect of material condition, or microstructure, on the transition velocity, specimens of this same material (S.A.E. No. 1035 steel) were tested in the quenched-and-tempered condition. The results compared with those from the annealed condition are shown in Fig. 7. It will be noted that this further heat treatment has raised the transition velocity from 30 ft. per sec. to 125 ft. per sec., with a similar although more rapid decrease in energy values, and increase in ductility, above this velocity.

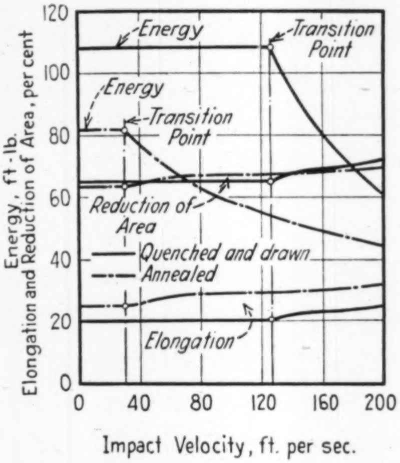

FIG. 7.—High-Velocity Tension Impact.
S.A.E. No. 1035 Steel.

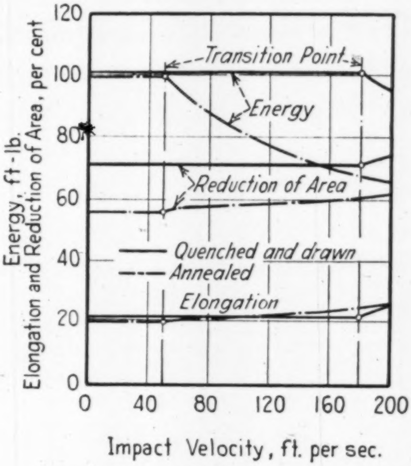

FIG. 8.—High-Velocity Tension Impact.
S.A.E. No. 2340 Steel.

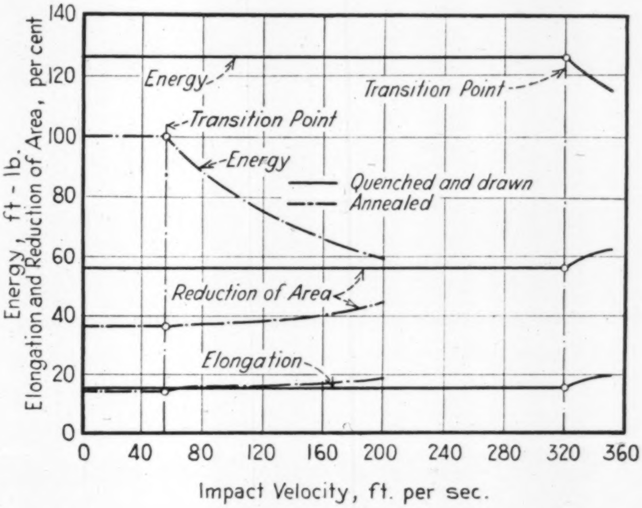

FIG. 9.—High-Velocity Tension Impact.
S.A.E. No. 4150 Steel.

Similar comparisons are shown in Figs. 8 and 9 for S.A.E. Nos. 2340 and 4150 steels. In the case of steel 2340, quenching and tempering has raised the transition velocity from 50 ft. per sec., in the annealed state, to 180 ft. per sec., and in the 4150 steel from 55 ft. per sec. to 320 ft. per sec. It will be noted that in each material the energy decreases and the ductility increases when tested at velocities above the transition point.

A preliminary examination of the microstructure at the fractured surfaces of specimens, ruptured both within and above the transition velocity, indicates that within the transition velocity, rupture takes place around the grains, and as the velocity is increased the form of break appears to change, first to 90 deg. across and then to 45-deg. shear through the grains. This change in the form of rupture was particularly evident in the silicon bronze and also in the S.A.E. No. 1035 annealed material. A more complete and thorough study is being undertaken, and until further evidence is accumulated no definite conclusion regarding this apparent phenomenon can be presented.

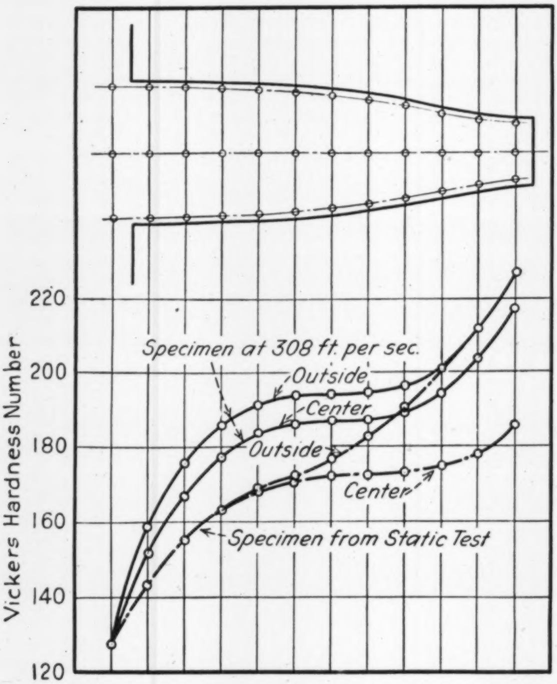

FIG. 10.—Showing Hardness Variations.
S.A.E. No. 1035 Steel.

To determine the work-hardening effect on material tested at within and above the transition velocity, specimens of 1035 steel annealed, one tested statically and the other at 308 ft. per sec., were sectioned and tested for hardness throughout the gage length, at the center and close to the outer surface. The results are shown in Fig. 10. It will be noted that the specimen tested at 308 ft. per sec., although requiring less energy for rupture but at the same time exhibiting greater ductility, shows considerably more work-hardening than the specimen tested under static conditions, indicating a correlation between work-hardening and amount of deformation.

While there appears to be no definite explanation of this phenomenon, it is suggested that, if resistance to deformation may be considered as the result of some form of energy transformation within the material, it is then possible to consider the transition velocity to represent the maximum rate at which complete energy transformation can take place. At higher velocities less resistance would then be offered, and therefore, less energy would be required for rupture and at the same time greater deformation would take place. Also, since it is apparent that time is required for this energy change within the material to be completed, the transformation once started might well continue to completion, even after rupture, thus resulting in the increased work-hardening noted.

DISCUSSION

The data presented conclusively show that high-velocity tests are essential to bring out the true dynamic properties of materials. From an extensive search of the literature pertaining to the subject of impact testing, it would appear that this investigation has revealed conclusively, for the first time, the fact that materials possess definite transition velocities which must not be exceeded if normal behavior is to be expected.

The transition velocity factor appears to offer a logical explanation of the many subsequent failures of materials which, from all the usual tests including the notched-bar impact test, were considered to be perfectly suitable for the service required. The full value of this significant factor to the engineer and metallurgist should be realized, when sufficient data have been obtained from tests of different materials, to prepare a series of transition velocity *versus* heat treatment diagrams. From such data it should then be possible to select the material and heat treatment best suited to meet service requirements involving a known rate of dynamic loading.

The results of this investigation further indicate that in the selection of materials for use under dynamic conditions, there are three significant factors which must be considered:

1. The transition velocity within which normal material behavior may be expected.
2. The ratio of total energy value to mass or volume of material involved.
3. The effect of surface irregularities, such as tool marks, size and form of fillets, etc.

The first two factors are considered to be directly concerned with the true dynamic properties of the material. Of these, the transition velocity can only be determined from high-velocity tests, and indicates the maximum safe rate for dynamic loading. The energy-volume ratio, however, can be evaluated from either the high-velocity or the static tests, and is considered to indicate, qualitatively at least, the mass or inertia effect on the total energy required for rupture.

The third factor is considered to be only indirectly concerned with the true dynamic properties of materials, since it merely indicates the general effect of the geometry, or form, of the specimen, when subjected to impact loading. From the fact that this general effect is revealed in the ordinary type of impact test, it would seem reasonable to conclude that much of the controversy and disagreement regarding the value of the impact test has resulted from the fact that the rate of loading is always within the transition velocity, and the results obtained, therefore, have at all times failed to disclose any evidence of the true dynamic properties.

In view of the transition velocity factor brought out by this investigation, it is quite apparent that total energy values alone are no criteria of serviceability, since a material may show a very high energy value, yet have a comparatively low transition velocity, which would make it entirely unfit for service conditions involving dynamic loading. It is, therefore, evident that these two factors must be considered together in the final analysis.

With regard to the increased ductility observed in the 1.0-in. gage length specimens when tested at velocities above the transition point, this condition has been found to apply to all materials except those in the austenitic or solid solution state. A few preliminary tests of such materials have indicated that, although the reduction of area increases, deformation is restricted to a shorter portion of the specimen gage length, resulting in decreased elongation values. This phenomenon, however, has not been sufficiently established to be presented at this time.

Although in the tests of No. 1035 steel annealed, the 0.1-in. gage length specimens showed decreased ductility when tested at velocities above the transition point, similar specimens from two different steels, which in the quenched-and-tempered condition had transition velocities of around 70 ft. per sec., showed in one case increased, and in the other decreased, ductility at higher velocities. The material showing increased ductility developed an elongation of over 100 per cent in the specimen tested at 200 ft. per sec. Since the steel which showed decreased ductility was also unsuitable for the particular service required, it is therefore quite probable that the ductility conditions above the transition velocity also have a significant bearing in the selection of materials for certain unusual service conditions.

While the data presented reveal certain heretofore unsuspected phenomena regarding the dynamic properties of materials, there are many factors which require further study and investigation before their full significance can be completely realized. The results obtained so far, however, are believed to clarify much of the mystery surrounding the ordinary type of impact test.

CONCLUSIONS

Although this investigation enters a new and unexplored field, the data presented appear amply to justify the following conclusions:

1. High-velocity tension-impact tests reveal the true dynamic properties of materials.
2. The transition velocity factor is a measurable value and therefore applicable for use in design.
3. The development of transition velocity *versus* heat treatment diagrams should facilitate the selection of the proper materials to meet service conditions involving a known rate of dynamic loading.
4. The ordinary type of impact test at only one velocity merely indicates the general effect of specimen geometry or form, and does not reveal the true dynamic properties of materials.

Acknowledgment:

The author wishes to acknowledge the indispensable assistance of Capt. R. K. Haskell, Ordnance Dept., in the design and development of the high-velocity impact machine, and of Lt.-Col. Earle B. Norris, Dean of Engineering, Virginia Polytechnic Institute, Prof. C. E. Fuller, Department of Theoretical and Applied Mechanics, Massachusetts Institute of Technology, and Lt. F. M. Humphries, Ordnance Dept., for their aid in developing the mathematical analysis presented and to Col. G. F. Jenks, Commanding Officer, Watertown Arsenal, whose active interest made this work possible.

APPENDIX

DERIVATION OF EQUATION FOR DETERMINING ENERGY REQUIRED FOR RUPTURE

Consider the energy measuring system and the kinetic energy system, both having a common axis of rotation, to be connected by the test specimen. During the process of rupture the kinetic energy system moves away from the energy measuring system at a velocity V producing at the same time an equal and opposite force at each end of the test specimen. This force is not constant but varies throughout the process of rupture. Since the work done on the test specimen is equal to the force F times the space through which it moves

$$\mathrm{d}E = F\mathrm{d}S \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

also $\mathrm{d}S = V\mathrm{d}t = r\omega\mathrm{d}t$ where ω is the angular velocity of the wheel with respect to the pendulum at any instant, and r is the radius from the axis of rotation to the point of application of the force.

Substituting in Eq. 1,

$$\mathrm{d}E = \omega Fr\mathrm{d}t \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

let $\omega = \omega_r - \omega_p$

where ω_r = angular velocity of wheel, and

ω_r = angular velocity of pendulum.

Substituting the value of ω in Eq. 2 and integrating

$$E = \int_0^t (\omega_r - \omega_p)\, Fr\mathrm{d}t \quad (3)$$

where E is the energy required to rupture the test specimen.

During the process of rupture, however, the pendulum accelerates and the wheel decelerates, therefore ω_r and ω_p are variables, the force F varies during the process of rupture, and the time t is a function of both the velocity and the total elongation of the test specimen. Consequently, Eq. 3 cannot be evaluated without knowing the change in angular momentum of the pendulum and the change in kinetic energy of the wheel.

Since the initial angular velocity of the pendulum is zero, the change in momentum may be expressed as follows:

$$\int_0^t Fr\mathrm{d}t = I_p\omega_p \quad (4)$$

where I_p = moment of inertia of the pendulum. Considering the change in kinetic energy of the wheel:

$$KE = \tfrac{1}{2} I_2 (\omega_1^2 - \omega_2^2) \quad (5)$$

where I_2 = moment of inertia—wheel and tup, horns out
ω_1 = initial angular velocity—wheel and tup, horns out, and
ω_2 = final angular velocity—wheel and tup, horns out.

Since the energy removed from the wheel goes to rupture the test specimen and move the pendulum, it is evident that the energy required for rupture may be found by determining ω_1, ω_2, and the kinetic energy imparted to the pendulum.

Let ω_0 = angular velocity of wheel at the instant the horns are released. Since the centers of gravity of the horns and counterweight move away from the axis of rotation, the moment of inertia of the wheel is increased. Also the tup must be accelerated to the velocity of the wheel before the process of rupture can take place. From the law of conservation of angular momentum,

$$I_1\omega_0 = I_3\omega_1 + \frac{lr^2}{g}\omega_1 \quad (6)$$

where I_1 = moment of inertia—wheel without tup, horns retracted,
I_3 = moment of inertia—wheel without tup, horns out, and
l = weight of tup.

then

$$\omega_1 = \frac{I_1\omega_0}{I_3 + \dfrac{lr^2}{g}} \text{ or } \frac{I_1\omega_0}{I_2} \quad (7)$$

which represents the angular velocity of the wheel at the instant the process of rupture begins.

Knowing the initial angular velocity of the wheel and the change in momentum produced during rupture, the final angular velocity may be calculated. However, since the force acting on the wheel and tup is the same as that which acts on the pendulum, the time it acts must be the same and the same change in momentum must have been produced in each.

The change in momentum of the pendulum was from Eq. 4 equal to $I_p\omega_p$. Then

$$I_2\omega_1 = I_2\omega_2 + I_p\omega_p \quad (8)$$

from which

$$\omega_2 = \omega_1 - \frac{I_p}{I_2}\omega_p \quad (9)$$

Substituting the values of ω_2 from Eq. 9 and ω_1 from Eq. 7 in Eq. 5:

$$KE = \tfrac{1}{2}\, I_2 \left[\left(\frac{I_1\omega_0}{I_2} \right)^2 - \left(\frac{I_1\omega_0}{I_2} - \frac{I_p}{I_2}\,\omega_2 \right)^2 \right] \qquad (10)$$

This equation represents the total kinetic energy change in the wheel. It is evident from Eq. 3 that the change in energy of the wheel concerns the work of rupturing the test specimen and also the energy imparted to the pendulum since

$$\int dE = \int_0^t \omega_r F r dt - \int_0^t \omega_p F r dt$$

where $\int \omega_r F r dt$ is the energy change in the wheel and $\int \omega_p F r dt$ is the energy imparted to the pendulum. Therefore, by subtracting the energy imparted to the pendulum from Eq. 10, the energy required to rupture the test specimen may be determined. From Eq. 10:

$$E = \tfrac{1}{2}\, I_2 \left[\left(\frac{I_1\omega_0}{I_2} \right)^2 - \left(\frac{I_1\omega_1}{I_2} - \frac{I_p}{I_2}\,\omega_2 \right)^2 \right] - E_p \qquad (11)$$

but

$$E_p = \tfrac{1}{2}\, I_p \omega_p^2$$

then

$$E = \frac{I_1\omega_0 I_p \omega_p}{I_2} - \tfrac{1}{2}\,\frac{I_p^2\omega_p^2}{I_2} - \tfrac{1}{2}\, I_p\omega_p^2 \qquad (12)$$

which simplifies to

$$E = \frac{I_1\omega_0 I_p \omega_p}{I_2} - \tfrac{1}{2}\, I_p\omega_p^2 \left(\frac{I_p + I_2}{I_2} \right) \qquad (13)$$

but

$$\omega_p = \sqrt{\frac{2LR(1 - \cos\theta)}{I_p}} \qquad (14)$$

where L = weight of pendulum,
R = radius to center of gravity, and
θ = angle of pendulum swing

Substituting the value of ω_p from Eq. 14 in Eq. 13:

$$E = \omega_0\,(1 - \cos\theta)^{\frac{1}{2}} \left[\frac{I_1}{I_2} \sqrt{2LRI_p} \right] - (1 - \cos\theta) \left[LR \left(\frac{I_p + I_2}{I_2} \right) \right] \qquad (15)$$

This equation determines the actual amount of energy required to rupture the test specimen, the only variables for which are obtained from tachometer and dial indicator readings for each test, and since the terms in brackets are constants of the machine the equation simplifies to the following:

$$E = \omega_0(1 - \cos\theta)^{\frac{1}{2}} K_1 - (1 - \cos\theta) K_2 \qquad (16)$$

DISCUSSION

MESSRS. D. S. CLARK[1] AND G. DÄTWYLER[2] (*presented in written form*).–The work that has been done at the Watertown Arsenal by H. C. Mann, as evidenced by his paper of last year[3] and the one now under discussion, is a real contribution in the field of impact testing. It has been our opinion for some time that the tension-impact test is of far more value in determing the relative impact resistance of materials than the customary notched bar tests now in common use. The present paper substantiates this view.

The conception of the transformation of energy as indicated in the present paper and brought forth in the paper of last year is not at all clear to us. A clear statement of the theory of energy transformation, we believe, would clarify the situation.

One of the points in the theory of conversion of internal energy that is questionable to us is Mr. Mann's inference that the amount of transformed energy is manifested by the amount of work hardening and that the increased elastic strength which is proportional to the change in sectional area is due to plastic deformation with its work hardening effect. It is difficult to see how this is possible, because the increased strength in the least cross-section is due, in a large degree, to the notch effect of the neck, as shown in the accompanying Fig. 1. These curves were obtained with two specimens, one of which was straight; the other had a neck machined to conform to the shape of a specimen just before failure occurs. The initial diameter of the cylindrical specimen was approximately the same as the original diameter of the necked specimen in the least diameter. True stresses are plotted against percentage reduction in area in the least cross-sections; that is, for the same amount of local deformation.

In last year's paper Mr. Mann showed that the impact value below the critical velocity is equal to the energy absorbed in slow static test as given by the area under the engineering curve ("external energy") plus the "internal energy." Now, according to the statement in the present paper, time is required for conversion of this internal energy. As the velocity of impact increases this time is denied and therefore one would expect the impact value above the critical velocity to drop to the energy given by the engineering curve. Actually the energy reaches a much lower value than this, although the elongation remains constant or even increases. This would indicate that the elongation is produced in the region above the critical velocity by much smaller forces than under static conditions.

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[1] Instructor of Mechanical Engineering, California Institute of Technology, Pasadena, Calif.

[2] Research Fellow in Aeronautics, California Institute of Technology, Pasadena, Calif.

[3] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

The curves shown in Fig. 10 of the present paper, giving hardness variations throughout the specimen, indicate a much more uniform plastic deformation in the necked portion under impact than under static loading. This effect may be attributed to inertia forces which tend to unify the stress distribution over the cross-section. This opinion could be further substantiated by measuring the hardness of specimens which have been broken at different impact velocities.

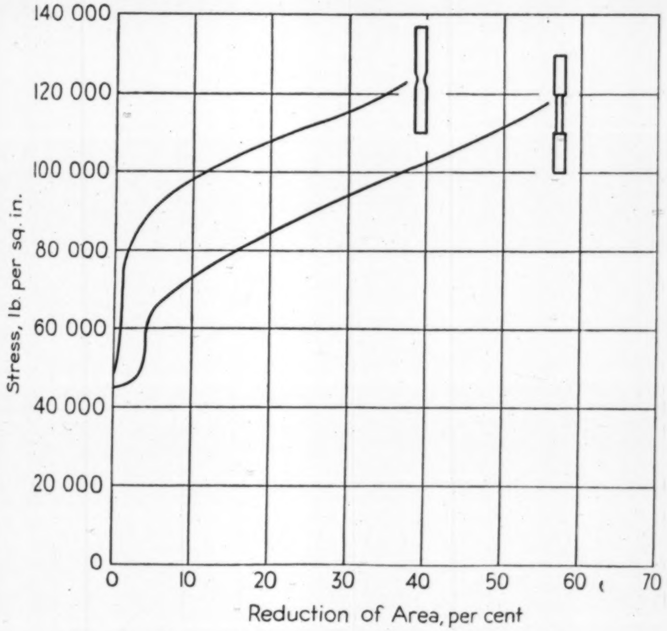

FIG. 1.—Stress - Reduction of Area Curves for Tension Impact Tests.

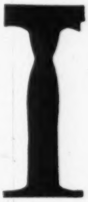

FIG. 2.—Tension-Impact Specimen.

It should be mentioned that this is not the first time the fact has been revealed that materials possess definite transition velocities which must not be exceeded if normal behavior is to be expected. Max Moser[4] in 1924 showed results which proved the existence of such a limiting impact velocity.

We would be interested in knowing whether or not in these investigations any double necking in the specimens was obtained. In our own work on tension impact we consistently found this phenomenon to occur with impact velocities between 60 and 250 ft. per sec. The accompanying Fig. 2 shows such a specimen (mild steel original size 1 in. by $\frac{1}{4}$-in. diameter). Increasing the radius of the fillets had no effect upon this phenomenon.

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[4] Max Moser, "Die Ergebnisse des Kerbschlagversuches," *Kruppsche Monatschefte*, Band 5, pp. 48–58 (1924).

MR. H. F. MOORE.[5]—In this paper Mr. Mann has made an excursion into what is almost unknown country—the field of the kinematics of stress. Nearly, if not quite, all our attempts to treat the question of "impact" or rapidly applied stress, as given in our standard texts on mechanics of materials, are based on the assumption that the general plan of stress distribution is just the same as it would be under some static load, and that the strength of our materials under rapid load does not vary seriously from the strength under static load. Mr. Mann has attacked this problem of strength under rapidly applied load from the side of the resistance of materials, and has brought out the fact that, especially above some critical speed of application, the resisting energy of a material may be greatly changed from its value for slowly applied load.

Approaching the problem from the side of the kind of stress distribution set up under rapid load there is some evidence that this distribution differs in kind as well as in magnitude from that set up under slowly applied load. The late Wilifred Campbell in his studies of stress in turbine disks has shown how interference between waves of stress may cause crests and nodes of stress which are entirely unaccounted for by considering static conditions. Recently at the University of Illinois, R. N. Arnold, a visiting graduate student on a (British) Commonwealth traveling fellowship, has been making an experimental and mathematical study of the elastic curve of a simple beam when struck by a falling weight. He has found that due to the inertia of the beam, the shape of the elastic curve when struck by a falling weight is of a distinctly different type from that assumed when statically loaded at the center of the span.

Mann has made a pioneer contribution in this field of kinematics of stress and strength of materials, and it is to be hoped that his paper will inspire others to explore this unknown country.

MR. H. I. HANSEN[6] (*by letter*).—In the paper by Mr. Mann, there are some noteworthy ideas, at the same time ideas quite difficult to understand or explain—for instance, deforming and rupturing a test specimen with an extremely small amount of energy. The question arises—is this something new and startling, or is it misinterpretation of the reading of the angle θ. Hang a heavy steel rod up as a pendulum and it is possible to move it with a slender lead pipe; but hit it a hard blow and the lead pipe will wrap itself around the steel rod—deformation without much movement of the measuring device. Try to push a door shut with the little finger—it does not hurt, and there is little sensation due to the reaction; but take a swing at the door, and the result will probably be a broken finger and a feeling of the door pushing back. This roughly illustrates the reaction of the pendulum in the testing machine.

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[5] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[6] Mechanical Engineer, A. O. Smith Corp., Milwaukee, Wis.

It is true that the available energy is $E_A = \frac{1}{2} I_2 (\omega_1^2 - \omega_2^2)$.

This energy is used (1) for the acceleration of the tup and pendulum, E_1 and (2) for the deformation of the test bar and thermal changes E_2, so the equation can be written

$$E_A = E_1 + E_2 = \tfrac{1}{2}I_2(\omega_1^2 - \omega_2^2)$$

or

$$E_2 = \tfrac{1}{2}I_2(\omega_1^2 - \omega_2^2) - E_1$$

If the tup is light and the pendulum does not show any appreciable movement (heavy anvil) $E_1 \sim 0$ which gives

$$E_2 = \tfrac{1}{2}I_2(\omega_1^2 - \omega_2^2)$$

The change of angular velocity could be checked with a stroboscope and the energy calculated from the observations. In this case, there would be little chance for energy loss to escape detection.

Equation 16 of Mann's paper, $E = \omega_0(1 - \cos\theta)^{\frac{1}{2}}K_1, - (1 - \cos\theta)K_2$, is based on D'Alembert's principle, and does not account for the actual deformation work, nor for thermal changes in the test specimen. Looking at the picture of a fracture at 200 ft. per sec. it is evident that a deformation of the test specimen has taken place and energy utilized, although it does not agree with Eq. 16. It is generally understood that an impact fracture of a brittle material shows very little or no energy absorption, that is, no deformation of the test specimen. (We are dealing here with a plain bar.) Making the test in slow motion, so to speak, it is possible to observe the different stages of impact. During the first part of the impact between the horns and the tup, the elongation of the test bar follows Hooke's law (elastic), secondly, a deformation takes place (plastic)—this is in harmony with Mann's curves, and it is interesting to note that as the point of transition velocity is passed and the energy decreases, the elongation, or in other words, the deformation increases—then rupture takes place.

$E - E_1 = E\dfrac{M_2}{M_1 + M_2}$ is the equation for the available energy in impact where one body, M_2, has the velocity 0.

Let M_2 be large compared to M_1—which gives, for no appreciable movement of M_2, $E_1 \sim 0$, and the available energy is used for deformation—as in drop forging. Let M_2 be small compared to M_1 and E_1 is large, but $E - E_1$ small, and the available energy is used for change of velocity of M_2—as in pile driving.

In the first case, the anvil did not move, but energy was utilized. Again

$$E - E_1 = \frac{M_1V_1^2}{2} - (1 - K^2)\frac{M_1M_2V_1^2}{2(M_1 + M_2)}$$

where V_1 = velocity of M_1 before impact, and
K = impact factor.

This factor is a function of the velocity but not a straight line—higher velocity, more deformation and correspondingly smaller K. For a perfectly elastic material, $K = 1$; for a perfectly plastic one, $K = 0$.

There should be no difficulties in an analogy with the impact as it takes place in Mann's machine, inasmuch as the elongation curve indicates this factor. The energy curve, from the transition velocity and on, would possibly show an increase in energy absorption (this must necessarily be confirmed by tests). Hoyt mentions in his recent articles on Notched Bar Testing, second installment under Variables—the observations of Hadfield and Main[7] that well-made, properly heat-treated steel remained tough even at bullet velocities of 600 m. per sec. and that the trend was towards higher impact value for increased velocities.

The mechanics of impact are brought into the discussion in the hope of getting a better understanding of the test. There is in this writer's mind some doubt as to the correctness of the way of measuring the available energy or the energy absorption, and more light on this point will bring out the value for the mechanical engineer.

MR. M. O. WITHEY[8] (*presented in written form*).—The author and his associates are to be congratulated for this very significant contribution to knowledge of the behavior of metals when stressed at high velocities. The marked differences in the rates at which the energies of rupture of the various metals tested diminish as the velocity is raised above the transition value are important and further studies correlating these findings with structure should furnish data of much value in design. The evidence presented on the magnitude of the transition velocities and the corresponding energies of rupture should be supplemented by more statistical data concerning the number of specimens and heats represented in the diagrams. Also there appear to be no data in the paper to substantiate the so-called second and third significant factors mentioned in the synopsis. Would it not be preferable to report the energy values in terms of unit volume based on the volume of metal in the middle portion of the specimen?

(*By letter*).—The above was written after a hasty review of the paper. A more careful scrutiny shows that the theory for evaluating the test results fails to account for the inertia effects of the specimen. These effects increase with the square of the velocity and if included would materially change the shapes of the right-hand portions of many of the energy-velocity curves.

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[7] S. L. Hoyt, "Notch Bar Testing," *Metals and Alloys*, January, 1936.
R. A. Hadfield and S. Main, *Proceedings*, Inst. Civil Engrs. (London), Vol. 221 (1920).

[8] Professor of Mechanics, University of Wisconsin, Madison, Wis.

MR. E. B. SMITH[9] (*by letter*).—This paper deals with a very complex subject and seems only to add confusion rather than to clarify any interpretation of the impact test. The author secures his results from a testing machine so designed that it may give misleading results.

The dimensional details of the testing machine are not given, so definite computations cannot be made. The free swinging pendulum can move only as a result of the force applied to it through the test specimen. At low velocities of the wheels and comparatively slow strains in the test specimen the pendulum will swing and give some indication, but at some sufficiently high wheel velocity the acceleration of the tup and head end of the specimen will be great enough to rupture the specimen in tension even though it were not fastened to the pendulum. This results from the mass of the tail end of the specimen and the force necessary to accelerate it to the velocity of the head end. The external force at the two ends of the specimen are opposite but are not equal when forces are dynamic and are acting under the influence of acceleration.

To illustrate what actually happens in one of these tests: Assume the elongation to be about 0.02 ft., and the weight of the specimen to be about 0.224 lb. (which are average values indicated in the paper). The velocity of the tup and head of the specimen increases from zero to some value depending on the speed of the wheels; variation is not linear but is assumed to be. Then at the low velocity of 30 ft. per sec. the acceleration of the tup will be approximately 22,500 ft. per sec. per sec. when rupture occurs, which in order to accelerate the dead end of the specimen to the same velocity will produce a force of only about 80 lb. This will not be sufficient to break the specimen, and since the acceleration cannot be changed in this single trial the additional force must be contributed by utilizing the mass of the pendulum to which the specimen is attached. This will cause the pendulum to swing and give a reading on the dial. But if the velocity for the test has been 200 ft. per sec. the acceleration would have been about 1,000,000 ft. per sec. per sec. with a resulting force in the specimen of about 3480 lb. This for the bronze specimen (Fig. 3) would have been sufficient to break the specimen even if it had not been attached to the pendulum, hence the pendulum will not swing and indicate any energy value. The author interprets his test results to indicate that the higher the velocity of force application the less the energy required for rupture. If he had made the provision of determining the initial and final energy in the revolving wheels of his testing machine he would have gotten more nearly the correct result.

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[9] Professor of Mechanical Engineering, College of the City of New York, New York City.

The author's mathematical analysis of his testing machine and results has the major error of not including the weight of the specimen in his Eq. 8. As indicated above this weight of the specimen will have a great effect on the result when the test is made at the higher velocities. The effect increases with the square of the velocity, and is sufficient to account for the drop in the energy-velocity curves.

MR. M. F. SAYRE[10] (*by letter*).—Mr. Mann in his work on high velocity tension-impact tests has done two distinct things. First, he has developed a form of impact testing which is much more logical than preceding methods in that he has separated the effect of speed of loading upon strength from other conflicting factors, and by isolating it made it easy to measure. The device used is obvious enough, once it has been suggested, but that by no means detracts from his credit for being the first to suggest it. In so far as Mr. Mann's experimental work and his conclusions can be verified, and he has already developed a strong *prima facie* case, the result would seem to be that the older forms of impact testing will be shoved into the background, while two new forms of test will take their place, one being Mr. Mann's test for effect of speed of loading, and the other a test yet to be developed, to measure separately what might be called a notch-concentration factor, which in part is the other component of our present impact testing.

Another, and much more important, thing which Mr. Mann has done, is to call to our attention a definite, clear-cut, effect of speed of loading upon strength characteristics. This is not an altogether new effect, but it is an effect which has not had the study which it deserved. Easy penetration of bullets through oak bulkheads has been treated as due simply to the tremendous energy present in the bullet, and not as possibly partly due to the reduced resistance to high-speed pressure inherent in the oak itself. Stories of pine planks driven through tree trunks, or of feathers driven into the pine planks, during tornadoes have been treated as old wives' fables. From a more immediately practical viewpoint, Mr. Mann's work seems likely to be of service in explaining many a previously inexplicable case of failure under service conditions both in Mr. Mann's own service, the U. S. Ordnance Division, and in other industries like the automotive industry, where high-speed motion or high-speed impact are likely service conditions.

Something more may possibly be done than has been done by Mr. Mann in analyzing conditions during the high-speed impact tension test itself, and in suggesting possible theories for the observed effects, not as proved but simply for discussion.

In Mr. Mann's apparatus the entire fracture takes place during a surprisingly short time. Assume, for example, an initial speed of impact of

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200 ft. per sec. and a total elongation of 0.3 in. in the 1-in. gage length specimen used. The forward speed of the lower end of the pendulum after the rupture would be of the order of magnitude of 5 to 10 ft. per sec. so small comparatively that it might almost be considered as stationary during the rupture. The horns would have dropped in velocity to possibly 180 ft. per sec. (exact values could of course be computed knowing the machine dimensions), giving a mean relative velocity during rupture, of horns with respect to pendulum of about 185 ft. per sec. Rupture must have occurred during the time required to travel 0.3 in. at that speed, or 0.000135 sec.

Four stages may be distinguished during this process of rupture: (1) the elastic stage, prior to beginning of plastic flow; (2) the period of elongation without increasing load (horizontal portion of the stress-strain curve next the yield point in steel); (3) the period of increasing load, prior to necking; and (4) the period after necking begins, with load decreasing.

At the speeds of loading used by Mr. Mann, the behavior during the first stage is widely different from that in the ordinary tension test. On impact of the horns with the tup, a wave of stress passes through the tup toward the specimen moving with the speed of sound in steel, about 203,000 in. per sec. ($=\sqrt{Eg/w}$, where w = density, lb. per cu. in.). On reaching the test specimen, part of this wave is reflected and the balance travels through the specimen at the same speed. If the effect of deflection in the tup is neglected, the magnitude of this initial stress wave in the reduced portion of the test specimen may be easily computed.

Let V_s be the wave velocity, V_t the forward velocity of the tup after being struck by the horns, E the modulus of elasticity of the specimen, and S the resulting stress. In dt seconds the wave will move forward $V_s dt$ inches. The resulting elongation of the specimen will be $\frac{S}{E}V_s\ dt$ inches. Ignoring plastic yield, this elastic elongation must equal the forward movement of the tup during the same period, or $V_t\ dt$. Therefore, $S = E\ V_t/V_s$.

For a steel test specimen, with tup brought up in 1/203,000 sec. to a speed of 200 ft. per sec. the front of the stress wave in the specimen would start down the specimen with an intensity of $30{,}000{,}000 \times 200 \times 12/203{,}000$, or 355,000 lb. per sq. in. It will travel down the 1-in. gage length in 0.0000049 sec. From the far end of the test specimen, a wave of the same velocity but of smaller stress intensity travels up the pendulum, while a reflected wave travels back through the test specimen. If by this time plastic yield has not come into play to relieve conditions, this stress wave will continue to reverberate through the specimen, the stress increasing by somewhat less than 355,000 lb. per sq. in. at each reflection, until an actual tension failure without plastic yield occurs. This may be what happened in the case of the fine crystalline fracture directly at the middle of the 0.1-in. gage length, described at the bottom of page 90 of the paper. Usually, of course, plastic yield intervenes, furnishing the necessary elongation without calling for these high stresses, so that the stress wave will be damped out in two or three cycles. The crest of the wave probably starts out with the full computed height as already given, so that momentarily a portion of the specimen is stressed elastically to far above its normal elastic limit, but the effect of plastic yield will be to cause a rapid drop in the crest height as the wave crest moves along the specimen.

The experimental results given by Mr. Mann show that up to his transition velocity, the energy absorbed in a high velocity tension break is essentially the same as for a static tension break on an equivalent specimen. This would suggest that except for this first, momentary stage, the velocity has but little effect upon the stress-strain relationship, so that up to the transition velocity, a stress-strain curve, if one could be obtained on Mr. Mann's machine, would differ but little from that given by the usual low-speed tension test. Experimental verification of this statement would be highly desirable.

Beyond the transition velocity, surprisingly, Mr. Mann's results indicate that the energy absorbed is less, rather than more, while the total elongation in general remains the same, or even increases. At any given unit strain (beyond the elastic stage) the stress at very high speeds of test is therefore apparently less than for the static test. This differs from the results obtained in previous studies of the effect of speed, in which in general the stress for a given rate of strain in the plastic range, due to creep, is lower at very low speeds of test than at moderate speeds. I am here ignoring the effect of time lag in the testing machine itself, which has also been a factor in some of the moderate speed tests. It should be noted however that the highest speed of loading experimented with in these tests, using customary testing machines, is much less than the lowest speed of loading developed in Mr. Mann's machine, so that different physical phenomena are involved and the results are not really comparable.

A second surprising feature in Mr. Mann's work is his Fig. 6, which indicates that the value of the transition velocity is the same for specimens with 0.1 in. and with 1 in. length of reduced section. This if true immediately imposes an important restriction upon possible explanations for the decrease in energy absorption which occurs above the transition velocity. During stage 1, within the elastic range, the height of the wave crest is determined by tup velocity and is independent of the gage length, so that if this stage is the determining one, it is logical that the transition velocity should be the same regardless of gage length. During stage 4, nearly all the elongation occurring is concentrated within the neck, which is more or less the same in length regardless of gage length. For a given tup velocity, the rate of plastic yield per inch of length per second of time is therefore more or less the same, regardless of gage length. During the two intermediate stages, plastic yield is distributed over the entire length of the reduced section of the specimen, so that for a given tup speed, the rate of plastic yield per inch of length per second of time would be ten times as great for the specimen with square notch and 0.1 in. gage length as for the 1-in. gage length specimen. One would therefore naturally expect that, if the reduction in energy absorption above the transition point were to be the result of phenomena caused by exceeding some limiting rate of plastic yield, the transition velocity would be dependent upon, rather than independent of the gage length. This for instance would appear to rule out any interpretation based upon time lag in restrengthening due to strain hardening, which *a priori* would seem to be the more probable one, and throw the explanation upon some phenomena dependent upon the height of the initial stress wave in stage 1 of the rupture. Further experimental work which I understand is now under way by Mr. Mann will aid in clarifying these and other points.

Mention should be made of one disturbing factor which may affect the numerical accuracy of the energy formulas given in the Appendix. These formulas have been derived on the assumption that the flywheel, tup, and pendulum each acts as a rigid unit. Any absorption of resilient energy in these parts, or any kinetic energy of internal vibration, will affect the accuracy of these equations. At the high speeds of impact used by Mr. Mann, the rupture of the specimen takes scarcely more time than that required for the stress wave to surge up the pendulum 11 or 12 in. to the shaft and then back to the specimen. The resulting vibrations within the sturdy pendulum used will be of extremely small amplitude but may absorb a sufficient amount of energy to affect appreciably the amplitude of the pendulum swing and so to modify the numerical results obtained. This is not a question of resonance effects between the period of the blow and the period of vibration within the pendulum, but simply the question of the effect of an extremely sharp blow upon a potentially vibrant system. This possible source of error deserves, I believe, further study.

MR. H. C. MANN[11] (*author's closure by letter*).—In reply to the questions raised by Messrs. D. S. Clark and G. Dätwyler, which for the most part concern the paper of last year, the initial conception of energy transformation was presented as a possible theory to account for the obvious lack of proper interpretation of the load-strain curve. Further studies of this subject, including the recent works of Gough, Taylor, and C. F. Elam (Mrs. G. H. Tipper) on the deformation of crystals, indicate the conception of internal potential energy to be basically correct. A more complete discussion of this theory is being prepared for presentation by R. K. Haskell, Ordnance Dept., U. S. Army, which I believe will greatly clarify the subject.

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[11] Senior Materials Engineer, Watertown Arsenal, Watertown, Mass.

If a series of tests had been carried out of specimens having various lengths of gage section, it would have been found that until the gage length was shortened to approximately three times the section diameter, the true stress *versus* reduction of area curves would have been essentially the same, indicating that the increased strength is due to work hardening and not to the notch effect of the neck. When the gage length is shortened still further the closer proximity of the shoulders restricts deformation of the reduced section resulting in higher observed elastic strength values. For any given material condition, however, the true breaking stress will be found the same irrespective of the specimen gage length.

With regard to the "time" element for energy conversion—this factor was recognized in the paper of last year as noted in conclusion 2 which states: "The work involved in producing deformation depends on force, *time*, and space. . . ." The fact that at velocities above the transition point the energy values decrease and the ductility increases, indicates a definite change in the form of both the load-strain and potential curves, although it does not necessarily follow that the initial force is less than the elastic strength of the material. It is our belief that the force required to start deformation is the same under all velocity conditions, but that from this point to the break the normal force-space curve is radically different at velocities above the transition point. It is expected that this phenomenon will be brought out through oscillographic methods now being developed for this purpose.

As to reference to the work of Max Moser, it was not the intent of this paper in any way to detract from his most careful investigations. His work, however, was carried out at low velocities only and did not reveal definitely the transition point of any single material. His conclusions, with respect to this factor, were therefore based on assumptions only. That Moser recognized this fact is evident from his statement that, "It is true that for a correct determination of the limiting speed of impact the carrying out of several series of tests is necessary, as well as apparatus for this purpose." Since we have developed the necessary apparatus and have carried out the necessary series of tests, we consider it perfectly justified to state that this investigation has for the first time conclusively revealed these limiting velocity or transition conditions.

Regarding double necking of the specimens, in our initial tests this condition occurred with certain types of material when the specimen gage section was perfectly cylindrical. By tapering toward the center to an amount of approximately 0.005 to 0.010 in., double necking was entirely eliminated. This procedure in our opinion does not in any way influence the results other than to promote greater uniformity; in fact, from our experience, the tapering of all tension test specimens, whether for static or dynamic tests, is to be recommended.

The remarks of Moore and Withey are appreciated. Statistical data concerning the number of specimens, heats, etc., is available but was purposely omitted from this paper since it was felt that the curves and discussion were sufficient to bring out the value of high velocity tests. The second and third factors mentioned in the synopsis, while not primarily substantiated by the data presented, were implied from the results obtained and were considered of sufficient importance to be included.

The reporting of energy values in terms of unit volume would be preferable in cases where all tests were carried out with specimens of the same geometric form. In this investigation, however, the specimens did not have this similarity, and since under these conditions the unit values are not constant, it was thought advisable to report only total energy values.

The questions raised by Hansen and Smith can be best answered by stating that tests have been carried out at velocities within and above the transition velocity, in which the energy for rupture has been determined from the energy change produced in the rotating wheel and the values have been found to check in each case with those determined from the pendulum. Furthermore, specimens of 18 per cent chromium, 8 per cent nickel stainless steel and S.A.E. No. 3435 steel which, in the condition tested, showed transition velocities of approximately 6 and 15 ft. per sec., respectively, were ruptured in a 300-kgm. Charpy impact machine at velocities of from 5 to 28.5 ft. per sec., the lower velocities being obtained by decreasing the height of fall of the pendulum. The results showed in each case the same effect brought out in the paper: decreased energy values at velocities above the transition point.

Mr. Sayre's highly constructive remarks and suggestions for further research are most timely. It is readily admitted that there are many phases of this problem which at present cannot be conclusively analyzed, principally because of the lack of basic information as to the form of the force-elongation diagram at the various velocities. Appreciating the importance of this factor a research program has been initiated to obtain this information by photo-electric and oscillographic methods. This work, if successful, should produce conclusive evidence of the true conditions involved under dynamic loading.

SLOW-BEND AND IMPACT TESTS OF NOTCHED BARS AT LOW TEMPERATURES

By H. F. Moore,[1] H. B. Wishart[2] and S. W. Lyon[2]

Synopsis

Results are given of slow-bend and impact tests of various metals at temperatures ranging from ordinary room temperature to 40 deg. below zero Fahr. The correlation between results from slow-bend tests and from impact tests over this temperature range is not greatly different from that found by Petrenko[3] for ordinary room temperatures. The autographic slow-bend testing machine used is described. In a flexure test of a notched-bar specimen the friction of striking edge along the specimen seems to be a rather serious source of error, especially for specimens of metal so ductile that after the test they are not broken clean in two. Tension tests of notched specimens, both impact tests and slow-pull tests, are suggested as worthy of experimental study.

Introduction

In the development of the Charpy and Izod impact tests of notched bars it has gradually become evident that the test is significant as an indication of the effect of a notch in a given metal to a greater extent than it is as an indication of the behavior of metal under a very rapidly applied load, although both speed of testing and notch effect are factors in these tests. In 1925, Petrenko of the National Bureau of Standards made a study of comparative energy required to break Izod specimens when applied by the impact of a swinging pendulum and when applied in a Humfrey slow-bend machine by a slowly increasing "static" load.[3]

The following quotation from Petrenko's paper sums up one of the principal conclusions of his work:

> "Wherever the variation of the notched bar values is present in the specimens made of the same material, whether this variation is due to the shape of notch or to its position in respect to the rolling direction, or to the variation in the notched-bar properties of a material, the slow-bend test gives results comparable with those of the impact test, but it is less responsive to these variations than the impact test. By means of a bending moment diagram the slow-bend test gives some values which are related to the tensile yield point and to the tensile strength of the materials.
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> "It is, however, less convenient than the impact for the ordinary routine practice."

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[3] S. N. Petrenko, "Comparative Slow Bend and Impact Notched Bar Tests on Some Metals," National Bureau of Standards *Technologic Paper No. 289* (1925).

Object of Present Tests.—Recently opportunity was afforded the authors to make a series of tests at low temperatures using one of the cold rooms at Wright Field, Dayton, Ohio. The main object of these tests was the study of the behavior of rail steel at low temperatures, but while the rail steel tests were being carried on, it was thought worth while to carry on tests similar to Petrenko's on several different metals and to see whether for temperatures as low as −40 F. the same conclusion could be drawn which Petrenko drew from his tests at ordinary room temperatures.

TABLE I.—METALS TESTED.

Copper specimens cut from $\frac{1}{2}$-in. square rod; all others from $\frac{7}{16}$-in. square rod.

Metal	Treatment	Brinell Hardness Number
Steels:		
S.A.E. No. 3135, heat-treated........	Received hot-rolled, annealed at 1600 F., oil-quenched from 1540 F., drawn at 600 F., quenched in water.	325 (3000-kg. load)
S.A.E. No. 3135, cold-rolled..........	Received cold-rolled, tested as received.	248 (3000-kg. load)
S.A.E. No. 1020, heat-treated........	Received hot-rolled, water-quenched from 1650 F., drawn at 1200 F.	129 (3000-kg. load)
S.A.E. No. 1020, cold-rolled..........	Received cold-rolled, tested as received.	179 (3000-kg. load)
S.A.E. No. 1095, heat-treated........	Received hot-rolled, normalized at 1600 F., oil-quenched from 1475 F., drawn at 800 F.	363 (3000-kg. load)
Non-Ferrous Metals:		
60-40 Brass, cold-drawn..............	Received cold-drawn, tested as received.	120 (500-kg. load)
Copper, cold-drawn..................	Received cold-drawn, tested as received.	100 (500-kg. load)
Duralumin (17-ST)..................	Tested as received.	112 (500-kg. load)

TABLE II.—RESULTS OF SLOW-BEND TESTS AND IMPACT TESTS.

Each value listed is the average of three test results. Speed of cross-head of slow-bend testing machine, 0.4 in. per min.

Metal	Slow-Bend Tests Energy for Fracture, ft-lb.				Impact Tests Energy for Fracture, ft-lb.			
	70 F.	10 F.	−20 F.	−40 F.	70 F.	10 F.	−20 F.	−40 F.
Steels:								
S.A.E. No. 3135, heat-treated.........	8.90	7.27	7.27	6.78	11.90	7.80	8.70	8.50
S.A.E. No. 3135, cold-rolled..........	11.60	6.10	4.72	2.95	9.50	4.00	3.20	3.40
S.A.E. No. 1020, heat-treated.........	90.50[a]	97.30[a]	91.90[a]	94.20[a]	108.70[a]	117.70[a]	118.70[a]	119.30[a]
S.A.E. No. 1020, cold-rolled..........	46.80[a]	10.21	8.31	6.04	10.00	4.40	4.20	4.20
S.A.E. No. 1095, heat-treated.........	12.10	10.36	9.74	10.86	17.80	14.30	8.60	13.50
Non-Ferrous Metals:								
60-40 Brass, cold-drawn..............	7.71	8.96	8.91	9.50	12.30	14.90	16.20	15.00
Copper, cold-drawn..................	51.40[a]	56.27[a]	56.70[a]	57.60[a]	62.70[a]	73.40[a]	78.60[a]	75.80[a]
Duralumin, (17-ST), as received.......	13.00	13.57	13.37	13.82	18.10	18.90	20.10	19.60

[a] Specimen did not break in two.

MATERIALS, SPECIMENS, AND TESTS

Table I shows the materials chosen for study together with the treatment to which the metals were subjected. The metals were chosen as offering a fairly wide range, and the number which could be tested within the time during which the cold room was available limited the number of metals tested.

Figure 1 shows the form and dimensions of the specimen used. In all cases tests were made in triplicate, and in a few cases additional specimens were tested.

The impact tests were made on an Izod machine with a capacity of

120 ft-lb. The striking velocity was 11 ft. per sec. The slow-bend tests were made on the machine shown in Fig. 2. This machine was an Olsen machine originally designed for testing cast iron arbitration bars over a 12-in. span. It was modified for these tests by mounting a vise *C* for an Izod specimen *S*, as shown in the diagram at the upper left hand of

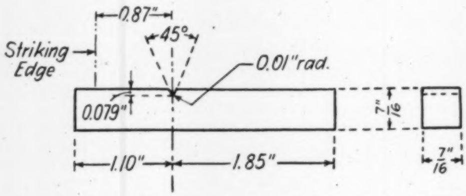

FIG. 1.—Specimen for Izod Impact Tests and Slow-Bend Tests.

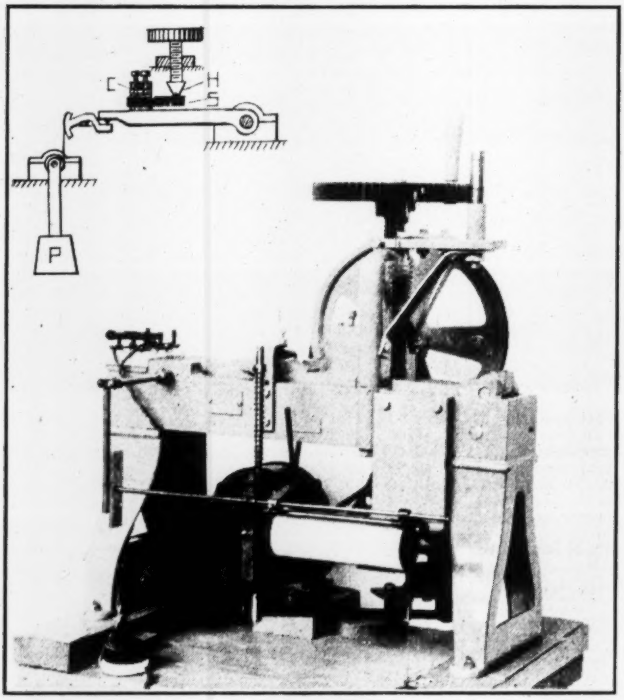

FIG. 2.—Testing Machine for Slow-Bend Flexure Tests of Notched Bars.

Fig. 2. The load-weighing mechanism consisted of a pendulum *P* whose motion moved a recording pencil along a drum. The downward motion of the cross-head *H* of the machine caused the drum to rotate, and an autographic load-deflection diagram was traced on a record sheet on the drum. The testing machine was driven by a motor, and the speed of the cross-head was approximately 0.4 in. per min.

RESULTS OF TESTS

The Izod impact machine registers directly the amount of energy necessary to break the specimen. With the slow-bend machine the energy necessary to break a specimen is measured by the area under a load-deflection diagram. The measurement of this area may be readily made by means of a planimeter. Table II shows the average results of the tests made, and Fig. 3 shows typical load-deflection diagrams for the slow-bend tests.

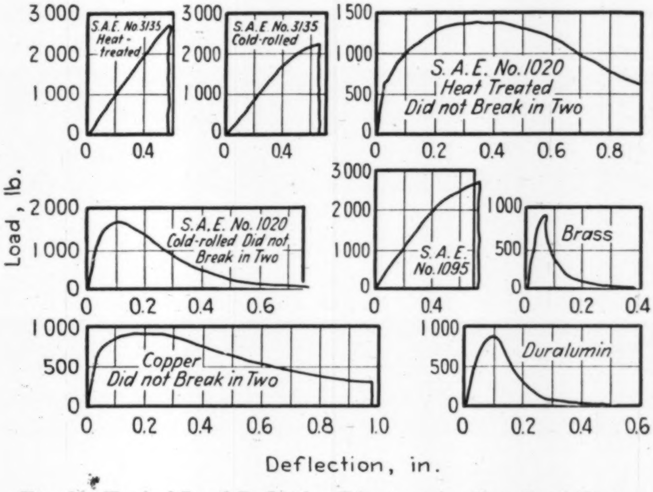

FIG. 3.—Typical Load-Deflection Diagrams for Slow-Bend Tests.

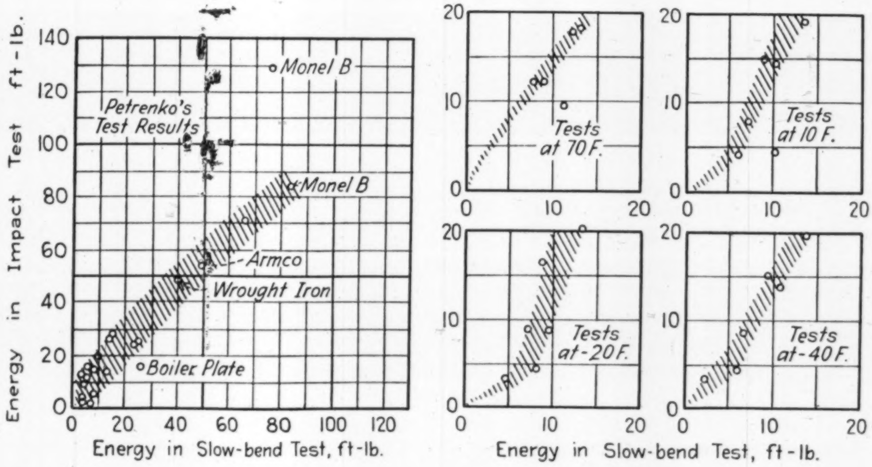

FIG. 4.—Results of Slow-Bend Tests and Impact Tests.

In drawing conclusions from these test results, special consideration was given to those specimens which did not break clean in two. With either the Izod machine or the slow-bend machine there is some energy used up in overcoming the friction of the striking edge of the machine as it moves *along* the specimen as the latter deflects before fracture. Evidently in the case of specimens which do not break clean in two this friction will be larger than in specimens which break clean in two. This friction is recorded in both the impact test and in the slow-bend test as part of the energy necessary to break the specimen.[4] It seemed best to the authors to disregard the test results from specimens which did not break clean in two, and hence the test results from the specimens of copper and of heat-treated S.A.E. No. 1020 steel, and from the specimens of cold-rolled S.A.E. No. 1020 steel at 70 F. have been disregarded in making comparisons of the test results from slow-bend tests with those from impact tests.

Omitting test results from specimens which did not break in two, Fig. 4 has been plotted. In Fig. 4 are shown an average of Petrenko's results, and an average of the test results obtained by the authors at Dayton at four different temperatures, 70, 10, −20, and −40 F.[5] These test results seem to agree in a general way with Petrenko's results, and to indicate that, for the metals tested, the slow-bend test gives results roughly comparable with those given by the impact test over a temperature range from 70 to −40 F. However, there is enough variation between the results of the two tests so that, if the metals are arranged in order of magnitude of energy for fracture, in general, they will not be arranged in the same order for Izod impact tests and for slow-bend tests. The following list shows such an arrangement for the metals tested.

Temperature, deg. Fahr.	Type of Test	Material[a]
70	Slow-bend	Duralumin, S.A.E. No. 1095, S.A.E. No. 3135CR, S.A.E. No .3135HT, Brass[b]
	Impact	Duralumin, S.A.E. No. 1095, Brass, S.A.E. No. 3135HT, S.A.E. No. 1020CR, S.A.E. No. 3135CR
10	Slow-bend	Duralumin, S.A.E. No. 1095, S.A.E. No. 1020CR, Brass, S.A.E. No. 35HT, S.A.E. No. 3135CR
	Impact	Duralumin, Brass, S.A.E. No. 1095, S.A.E. No. 3135HT, S.A.E. No. 1020CR, S.A.E. No. 3135CR
−20	Slow-bend	Duralumin, S.A.E. No. 1095, Brass S.A.E. No. 1020CR, S.A.E. No. 3135HT, S.A.E. No. 3135CR
	Impact	Duralumin, Brass, S.A.E. No. 3135HT, S.A.E. No. 1095, S.A.E. No. 1020CR, S.A.E. No. 3135CR
−40	Slow-bend	Duralumin, S.A.E. No. 1095, Brass, S.A.E. No. 3135HT, S.A.E. No. 1020CR, S.A.E. No. 3135CR
	Impact	Duralumin, Brass, S.A.E. No. 1095, S.A.E. No. 3135HT, S.A.E. No. 1020CR, S.A.E. No 3135CR

[a] CR denotes cold-rolled metal; HT denotes heat-treated metal.
[b] Specimens of S.A.E. No. 1020 cold rolled did not break clean in two.

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[4] In Petrenko's tests an attempt was made to evaluate this energy lost in friction by allowing the striking edge to slide over the specimens which had been cracked but not broken in two by the test blow or load, and noting the energy used up. His method is not explained in detail, but it seems to the authors of this paper that the friction-energy lost as the striking edge slides along an uncracked specimen would be decidedly greater than the friction energy lost when it slides along a cracked and bent specimen.

[5] It is to be noted that the graph of Petrenko's results is plotted to a different scale than the results of the authors' tests.

However, it is generally accepted that only rather large differences in impact values have much significance, and some of the order of metals in the above list would, in some cases, be altered by rather small changes in impact values or slow-bend values.

An examination of Table II shows that for the cold-rolled steels the slow-bend values were greater than the impact values; for the other metals the impact values were the greater. For brass and duralumin the energy for fracture increased with lowering temperature; for S.A.E. No. 3135; S.A.E. No. 1020 cold-rolled, and S.A.E. No. 1095 the energy for fracture decreased with lowering temperature.

TENSION TESTS OF NOTCHED SPECIMENS

The large friction-energy loss in flexure tests of notched bars of ductile metals raises the question of the use of a tension test of notched specimens, either a slow-tension test or a tension impact test. In a tension test this friction loss would be eliminated. As stated by Petrenko, the slow-bend test is less convenient to make than the impact flexure test. In the case of tension tests of notched specimens the slow-tension test would not necessarily be less convenient than the tension impact test, on account of the care necessary to be sure that the tension impact specimen shall be free from bending, and the comparative ease of adjustment of the slow-tension test specimen by means of spherical bearings. However this may be, the authors throw out the suggestion that tension tests of notched bars (both impact tests and tests under slow loading) seem worth while investigating.

Acknowledgment:

Acknowledgment is made to the U. S. Air Service at Wright Field, Dayton, Ohio. One of the cold rooms at the field was very generously placed at the disposal of the authors for these tests, in connection with low-temperature tests of railroad rail steel. Special acknowledgment is made for the help and suggestions of the staff of the Materials Branch at Wright Field, and especially to Mr. J. B. Johnson, the Director.

DISCUSSION

Mr. H. C. Mann[1] (*presented in written form*).—The data obtained from the static and impact tests of the Izod form of specimen are interesting. In considering the method used to determine the energy value of the static test and comparing the static and dynamic values (Table II), it is apparent that the authors have neglected to consider the fact brought out in a paper presented at the 1935 annual meeting,[2] that when deformation occurs during the rupturing of the test specimen the usual force-space diagram obtained from the static test does not indicate the total energy concerned and is always less than the energy value from the dynamic test. The data recorded for the heat-treated steels and the non-ferrous materials appear to confirm this fact and agree with the results of similar tests using the tension form of specimen, the static energy value being that represented by the usual force-space diagram without consideration of the deformation factor. It is believed that this fact rather than the friction element is the more logical explanation of the differences noted.

With regard to the cold-rolled material, it has been our experience that under dynamic test the behavior of cold-rolled non-ferrous materials is comparable with that of heat-treated steels. Cold-rolled steel however has been found to be very erratic and extremely sensitive to velocity effects. The results obtained by the authors are therefore not considered at all unusual.

The suggestion that the tension form of specimen for both static and dynamic tests is worthy of experimental study is most definitely concurred with. The tension form of specimen for impact testing has been advocated by the Watertown Arsenal Laboratory for the past several years as capable of yielding results of greater significance and less susceptible to minor variations than the flexure type of specimen.

It is hoped that the authors may duplicate this work using the tension form of specimen and considering the deformation factor in determining the static energy value.

Mr. H. F. Moore.[3]—Answering Mr. Mann's remarks concerning the loss of energy due to friction during the bending of ductile specimens, I would note that for the specimens which did not break clean in two, a rough estimate indicated that the friction loss might be as high as 20 ft.-lb.—a very appreciable amount. Since there was no means available of comparing this loss for slow bend tests with the loss for impact tests of the same material, it was judged best to discard all results of tests in which the specimens were not broken clean in two.

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[1] Senior Materials Engineer, Watertown Arsenal, Watertown, Mass.

[2] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

[3] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

As to the inclusion of a neglected internal energy factor in the tests, I would state that the basis of comparison used for the two tests was the external energy required to break similar specimens. The object of the paper is to seek an answer to the question of whether the results of slow-bend tests can be correlated with the results of ordinary Izod tests at low temperatures. This external energy is determined from the fall and the rise of the pendulum of the impact machine, and from the area under the load-deflection diagram in the slow-bend test. The unknown and variable friction loss in the tests of specimens which did not break clean in two seems to the authors to justify rejecting quantitative comparisons between slow-bend and impact tests of such specimens. The question of the variation of the "deformation factor" did not enter into this question of rejection of comparative results of such incompletely broken specimens.

DAMAGE AND OVERSTRESS IN THE FATIGUE OF FERROUS MATERIALS

By H. W. Russell[1] and W. A. Welcker, Jr.[1]

Synopsis

Fatigue studies were made on twelve ferrous materials covering a wide range of structure and properties. These studies include the stress-cycle curve to high stresses of conventional polished specimens and of specimens with a square notch. The damage produced by overstressing for a number of cycles insufficient to cause fracture was investigated with both polished and notched specimens.

It is possible to establish with some definiteness a zone on the stress-cycle diagram, above the endurance limit, and bounded by a "damage line," within which no damage is done to the specimen as indicated by its ability to run subsequently at the endurance limit for an indefinite number of cycles. A 0.35 per cent carbon steel showed almost no resistance to overstress, while notched specimens of wrought iron were remarkably resistant to damage by overstress, the damage line almost coinciding with the stress-cycle curve.

An attempt is made to find some correlation between the high stress fatigue properties and the results of tension and impact tests. No clear-cut relations are found. However, some insight is given into the mechanisms of notch formation and notch propagation.

Introduction

After all the studies of failure under repeated stress and all the work it took to bring fatigue testing to the point where any laboratory will report the same endurance limit on standard-radius polished specimens of identical material, it is disconcerting to find that of two materials with different endurance limits, the one with the higher endurance limit is not necessarily the one which shows the best performance in actual service under repeated stress. For example, based on laboratory tests wrought iron would scarcely be chosen for resistance to repeated stress, yet it is commonly used in railroad work for just such service, in spite of the laboratory evidence.

If one seeks to explain the difference between the laboratory evidence and that of service by taking some other property into consideration as well, ductility is a property that at once comes to mind, yet the performance of such materials as the cast Ford crankshaft, which is almost devoid of ductility, indicates that this is not a sufficient added criterion.

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[1] Chief Physicist, and Mechanical Engineer, respectively, Battelle Memorial Inst., Columbus, Ohio.

The suspicion arises that damping properties (the ability to absorb energy) are of paramount importance, but all the available data on damping have not yet brought much clarification to our ideas of the value of damping ability.

A possible clue to these discrepancies may be at hand if we approach the problem primarily from the evaluation of service conditions and realize that in many types of service it is possible for occasional high stress to be imposed upon the part in service, stresses that would cause early failure were they continually repeated, and would thus lead to prompt redesign of the part. If then, a primary criterion of suitability is the ability of a material to stand an occasional overload without showing signs of distress, and the endurance limit tells only that part of the story which relates to the normal service stresses, the tangle might be unravelled.

In the design of machinery and other structural elements subjected to repeated stress the conventional fatigue limit is a valuable guide in the selection of a suitable material. It has, however, long been recognized that materials are seldom subjected in service to exactly the conditions used in the conventional fatigue test and that this test may fail to predict performance completely. Furthermore, failure after a sufficiently long time can be tolerated in some cases,[2] and hence life at a given stress may be more important than the fatigue limit.

Experience with materials in service has indicated that as a rough approximation the endurance limit does classify materials as to their relative order of merit under various conditions. Thus, if one material has a substantially higher endurance limit than another, it will in general have a higher tensile strength, a higher resistance to cyclic stress combined with steady stress, a longer life at high cyclic stress, and a higher fatigue limit in the presence of notches. But, if the endurance limits are not widely different none of these generalizations may be true.

It is the purpose of this investigation to study testing methods and to secure data which may permit a more satisfactory choice of materials, and to clarify certain imperfectly understood peculiarities of materials which have been noted in service. It is also hoped that it may be possible to throw more light on the mechanism of fatigue in general.

In service the material may be overstressed (stressed above its endurance limit) all or a part of the time. Notches may be present to add to the complexity. It is not practical to duplicate in the laboratory all the combinations of conditions which may occur. But certain trends can be established.

Consider the effects of overstress for a number of cycles insufficient to cause fracture. From the meaning of overstress it follows that overstress continued for a sufficient number of cycles will cause failure. Hence it is obvious that the first portion of the stressing period has contributed to the failure. In this sense overstress will cause damage always.

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[2] J. O. Almen and A. L. Boegehold, "Rear Axle Gears: Factors Which Influence Their Life," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 99 (1935).

A group of specimens may all be given the same overstress and then the endurance limit of these overstressed specimens determined. Normally it would be expected that this endurance limit would be less than the endurance limit of virgin specimens and under certain conditions just this result is secured. But, somewhat surprisingly, under other conditions the new endurance limit is greater than the virgin limit. A new group of specimens may then be overstressed for some other combination of stress and number of cycles and an endurance limit of this group determined. This process could be repeated a number of times, giving the endurance limits of various conditions of overstress.

The above process would require much time and material, as not less than two specimens are needed for each condition of overstress. A considerable simplification which will yield less, but adequate, information can be made. A single specimen can be overstressed, and then tested at the endurance limit of the virgin material. If fracture occurs the endurance limit related to this condition of overstress must be less than that of the virgin material and the overstressing may be said to have damaged the specimen. If, however, the overstressed specimen does not fail after a considerable number of cycles (10,000,000) its fatigue limit is at least as great as the limit of the virgin material and hence the overstressing has not damaged it.

By making a number of such tests with different conditions of overstress it is possible to establish, on the stress-cycle chart, a line called the damage line which separates the region containing the conditions of overstress and number of cycles causing damage from the conditions in which no damage is done. Such determinations of the damage line were made by the authors for H. J. French[3] and reported by him in the Campbell Lecture of 1933. Brophy[4] and Moore, in discussion, have reported similar tests.

It is generally conceded that the endurance test is very sensitive to slight lack of uniformity in a lot of material. Duplicate specimens stressed the same may show large differences in the number of cycles for fracture. This "scatter" of the results leads to uncertainty in the endurance limit, or stated loosely, the endurance limit of one specimen may be different from that of a supposed duplicate. Thus a magnified "scatter" occurs in the damage line. Even in the absence of "scatter" the determination of the position of the damage line is time consuming. When scatter occurs the exact position of the damage line as indicated from tests of a moderate number of specimens is not located with certainty. Hence French chose to speak of the "probable damage line," meaning that the probable position of the damage line was given.

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[3] H. J. French, "Fatigue and the Hardening of Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21, pp. 899-946 (1933).

[4] G. R. Brophy, "Damping Capacity, a Factor in Fatigue," *Transactions*, Am. Soc. Metals, Vol. 24, pp. 154-185 (1936).

The stress-cycle curve of fatigue provides a criterion for failure under continued overstress. While a portion of this curve at stresses above the endurance limit is usually determined, its form is seldom discussed, and the tests are seldom carried to stresses causing failure in a moderate number of cycles.

TABLE I.—COMPOSITION AND HEAT TREATMENT OF MATERIALS.

Material	Composition, per cent										Heat Treatment
	Combined Carbon	Total Carbon	Silicon	Manganese	Sulfur	Phosphorus	Copper	Molybdenum	Chromium	Nickel	
No. 1. Cast iron	0.58	3.03	1.93	0.64	0.023	0.076	—	—	—	—	None
No. 2. Molybdenum cast iron	0.57	2.95	1.94	0.64	0.023	0.076	—	0.90	—	—	None
No. 3. Cast pearlitic malleable	1.30	1.66	0.89	0.70	0.030	<0.03	1.78	—	0.65	—	Ford crankshaft as received
No. 4. Malleable iron	—	2.24	1.02	0.25	0.072	0.169	—	—	—	—	50 hr. at 1650 F.
No. 5. Copper malleable	—	2.26	0.90	0.28	0.019	0.15	1.12	—	—	—	50 hr. at 1650 F.
No. 6. Copper malleable, precipitation-hardened											50 hr. at 1650 F., 1½ hr. at 1350 F., 3 hr. at 930 F.
No. 7. Copper cast steel	—	0.23	0.38	0.61	0.031	0.044	1.20	—	—	—	1 hr. at 1650 F., air cooled
No. 8. Copper cast steel, precipitation-hardened											1 hr. at 1650 F., 3 hr. at 930 F.
No. 9. Wrought iron A	—	0.017	0.122	0.004	0.019	0.084	(slag 2.24)				None (longitudinal)
No. 10. Wrought iron AT											None (transverse)
No. 11. Wrought iron B	—	0.024	0.112	0.034	0.018	0.102	(slag 1.22)				None (longitudinal)
No. 12. Wrought iron BT											None (transverse)
No. 13. Ingot iron	—	0.014	—	0.015	0.026	0.004	0.035	—	—	—	30 min. at 1725 F., air cooled
No. 14. 0.35 per cent carbon steel	—	0.35	0.19	0.55	0.030	0.016	—	—	—	—	Annealed at 1280 F.
No. 15. S.A.E. no. 4130 steel	—	0.36	0.23	0.61	0.014	0.017	—	0.18	0.70	0.16	1 hr. at 1600 F., oil quenched, 1 hr. at 1000 F.
No. 16. 18 per cent chromium, 8 per cent nickel, stainless steel	—	0.09	0.020	0.39	0.02	0.008	—	—	17.51	9.24	Annealed

The damage line, in part, determines the conditions under which occasional overstress will contribute to failure. If the range of stress is constantly varying from overstress to understress the damage line may be inadequate for predicting failure. However, a high resistance to damage by overstressing is almost certainly a desirable property in a material subjected to such conditions.

Overstress may readily arise in the presence of a notch. Even at the fatigue limit the true fiber stress, as derived from advanced elastic theory and photoelastic measurements, is greater than the fiber stress at the endurance limit of unnotched specimens. The portion of the stress-cycle curve at stresses causing failure in a moderate number of cycles is also of importance, and has been carried to failure in about 25,000 cycles in this investigation.

The discussion of the damage line for conventional specimens may also be applied to notched specimens. After overstressing a notched specimen at a selected stress and number of cycles the specimen is tested at the fatigue limit of the notched material.

The present investigation deals then with overstress as represented by the stress-cycle curves and damage lines of both conventional and notched specimens. Ferrous materials with a wide range of structure and properties were selected for test in order that any general relations between properties might be revealed, while at the same time false generalizations based on an insufficient number and variety of materials might be avoided.

MATERIALS INVESTIGATED

The composition and condition of each of the materials used in this investigation are given in Table I. Most of the cast materials were prepared for this investigation under laboratory conditions. Material No. 3 was machined from a Ford engine crankshaft. The wrought materials were made under commercial conditions. The wrought irons were rolled specially, and were possibly inferior to regular production. Material No. 14 was the K-20 material which is being extensively investigated by the Joint A.S.M.E.-A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals. Materials Nos. 15 and 16 were materials previously investigated,[5] but of which there was not sufficient material remaining for all the tests made on the other samples in the present investigation.

SPECIMENS

Rotating-beam fatigue testing machines of the R. R. Moore type were used. These normally operate at 1725 r.p.m. but when it was necessary to overstress a specimen for a small accurately known number of cycles, a reducing gear giving 69 r.p.m. was used.

The standard fatigue specimens were of the conventional type, but with a longitudinal radius of curvature which avoided the fillets at the ends. They were given a final polish in the longitudinal direction with No. 000 metallographic paper.

The notched fatigue specimens had the form used by Kommers[6] as shown in the insert in Fig. 1.

The tool for cutting the notch was carefully dressed, and care was taken to secure as sharp a 90-deg. notch as possible. The radius of curvature of the corner is not known but is believed to be less than 0.001 in.

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[5] H. W. Russell and W. A. Welcker, Jr., "Endurance and Other Properties at Low Temperatures of Service Alloys for Aircraft Use," *Technical Notes*, National Advisory Committee for Aeronautics, No. 381, Washington, June, 1931.

[6] J. B. Kommers, "The Effect of Under-Stressing on Cast Iron and Open-Hearth Iron," ***Proceedings***, Am. Soc. Testing Mats., Vol. 30, Part II, p. 368 (1930).

This type of specimen was selected as it provides an extremely severe notch effect. The diameter of the reduced section is readily measured with accuracy. The nominal stress for these specimens was computed by the simple beam formula on the reduced section.

TABLE II.—STATIC AND IMPACT PROPERTIES OF MATERIALS.

Material	Yield Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Breaking Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Charpy Impact, ft-lb.	
						Keyhole Notch	V-Notch
No. 1. Cast iron	—	44 700	—	nil	nil	1.0	0.9
No. 2. Molybdenum cast iron	—	50 500	—	nil	nil	1.0	1.0
No. 3. Cast pearlitic malleable	—	78 250	—	nil	nil	1.0	—
No. 4. Malleable iron	29 300	54 200	66 700	23.0	19.2	6.8	5.5
No. 5. Copper malleable	38 000	57 250	71 300	19.0	22.0	8.0	7.0
No. 6. Copper malleable, hardened	46 100	65 000	80 300	17.5	19.5	7.6	6.6
No. 7. Copper cast steel	57 500	88 100	149 500	30.5	53.8	18.7	12.4
No. 8. Copper cast steel, hardened	79 400	106 300	165 800	24.0	48.4	12.0	3.3
No. 9. Wrought iron A	29 900	47 050	50 800	15.0	16.0	42.4	16.6
No. 10. Wrought iron AT	28 600	46 750	55 100	17.0	16.3	6.5	5.1
No. 11. Wrought iron B	39 800	47 200	49 100	38.5	48.2	28.7	20.8
No. 12. Wrought iron BT	23 700	37 500	34 000	6.5	12.3	11.1	7.9
No. 13. Ingot iron	23 600	44 850	55 300	47.0	47.0	41.9	18.9
No. 14. 0.35 per cent carbon steel	42 700	68 000	142 600	39.0	66.3	30.1	15.9
No. 15. S.A.E. No. 4130 steel	99 400	125 000	—	15.9	61.5	15.9	—
No. 16. 18 per cent chromium, 8 per cent nickel, stainless steel	—	88 000	—	69.9	76.7	76.7	66.0

TABLE III.—FATIGUE PROPERTIES OF MATERIALS.

Material	Fatigue Limit, lb. per sq. in.		Fatigue Ratio, Fatigue Limit to Tensile Strength		Notch Resistance, Fatigue Limit (Notched) / Fatigue Limit (Standard)	Relative Stress at 25,000 cycles			
						Stress / Fatigue Limit		Damage Stress / Fatigue Limit	
	Standard	Notched	Standard	Notched		Standard	Notched	Standard	Notched
No. 1. Cast iron	20 000	17 000	0.45	0.38	0.85	1.60	1.40	1.5	1.1
No. 2. Molybdenum cast iron	28 000	20 000	0.55	0.40	0.72	1.50	1.47	1.04	1.1
No. 3. Cast pearlitic malleable	44 000	31 000	0.56	0.50	0.71	1.52	1.78	1.21	1.0
No. 4. Malleable iron	29 000	19 000	0.54	0.35	0.66	1.55	1.58	1.34	1.47
No. 5. Copper malleable	32 000	21 000	0.56	0.37	0.66	1.41	1.72	1.00	1.0
No. 6. Copper malleable, hardened	33 000	20 000	0.51	0.31	0.61	1.51	1.90	1.18	1.75
No. 7. Copper cast steel	49 000	28 000	0.56	0.32	0.57	1.53	1.82	1.14	1.07
No. 8. Copper cast steel, hardened	58 000	28 000	0.55	0.26	0.48	1.73	1.89	1.21	1.57
No. 9. Wrought iron A	30 500	20 000	0.65	0.43	0.66	1.57	1.70	1.03	1.47
No. 10. Wrought iron AT	28 000	19 000	0.60	0.41	0.68	1.46	1.69	1.20	1.32
No. 11. Wrought iron B	25 000	19 000	0.53	0.40	0.76	1.80	1.84	1.42	1.71
No. 12. Wrought iron BT	25 000	18 000	0.67	0.48	0.72	1.64	1.67	1.20	1.61
No. 13. Ingot iron	29 000	19 000	0.65	0.42	0.66	1.45	1.71	1.00	1.53
No. 14. 0.35 per cent carbon steel	35 500	20 500	0.52	0.30	0.58	1.41	1.85	1.01	1.17
No. 15. S.A.E. No. 4130 steel	65 000	—	0.52	—	—	1.40	—	1.12	—
No. 16. 18 per cent chromium, 8 per cent nickel, stainless steel	33 000	36 000	0.38	0.41	1.09	1.12	1.22	1.09	1.11

Conventional keyhole and 45-deg. V-notch Charpy impact specimens were used. The V-notch had the depth of the standard keyhole notch.

RESULTS

The static and impact properties of the materials are shown in Table II. The breaking strength, determined from the autographic record of the

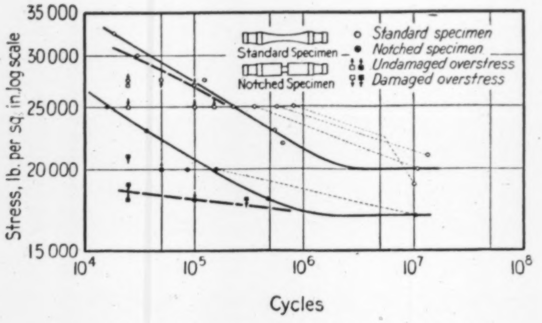

FIG. 1.—Fatigue Curves for Cast Iron No. 1.

The upper curve was determined with the standard specimen shown. The lower curve was determined with the notched specimen shown. Damage lines are given as heavy dashed lines.

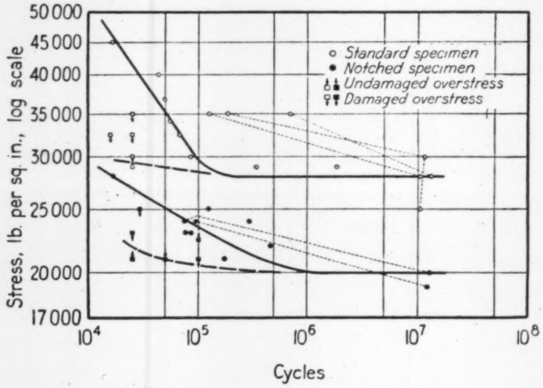

FIG. 2.—Fatigue Curves for Molybdenum Cast Iron No. 2.

Damage lines are given as heavy dashed lines.

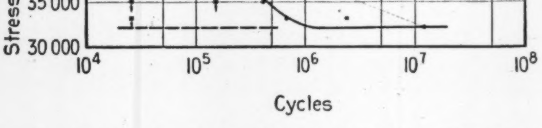

FIG. 3.—Fatigue Curves for Cast Pearlitic Malleable No. 3.

Damage lines are given as heavy dashed lines.

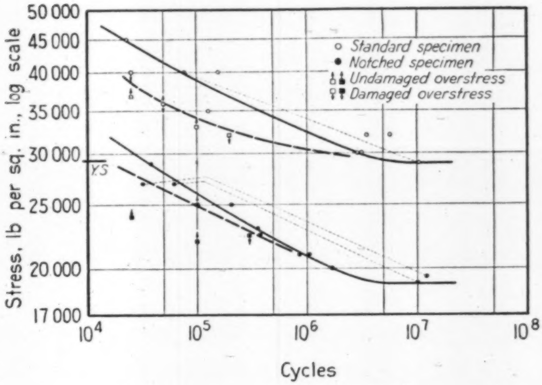

FIG. 4.—Fatigue Curves for Malleable Iron No. 4.
Damage lines are given as heavy dashed lines.

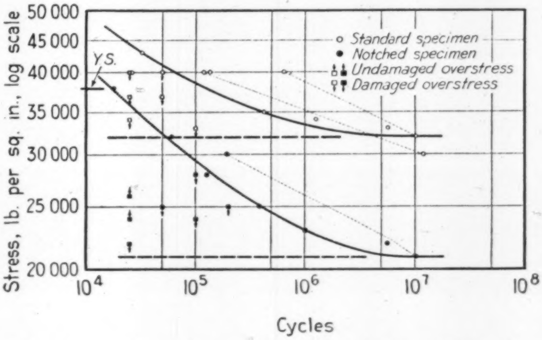

FIG. 5.—Fatigue Curves for Copper Malleable No. 5.
Damage lines are given as heavy dashed lines.

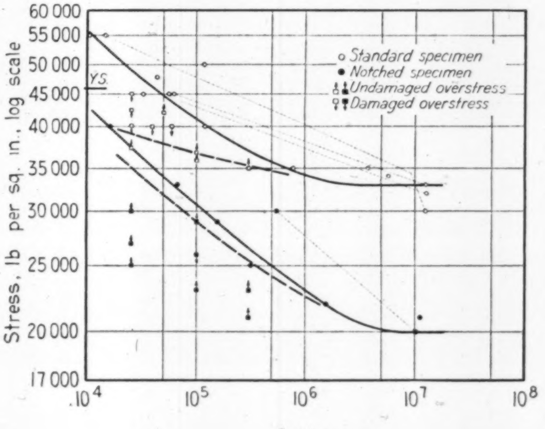

FIG. 6.—Fatigue Curves for Copper Malleable Precipitation-Hardened No. 6.
Damage lines are given as heavy dashed lines.

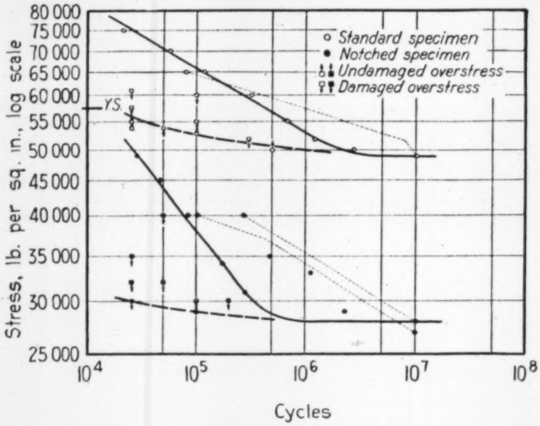

FIG. 7.—Fatigue Curves for Copper Cast Steel No. 7.
Damage lines are given as heavy dashed lines.

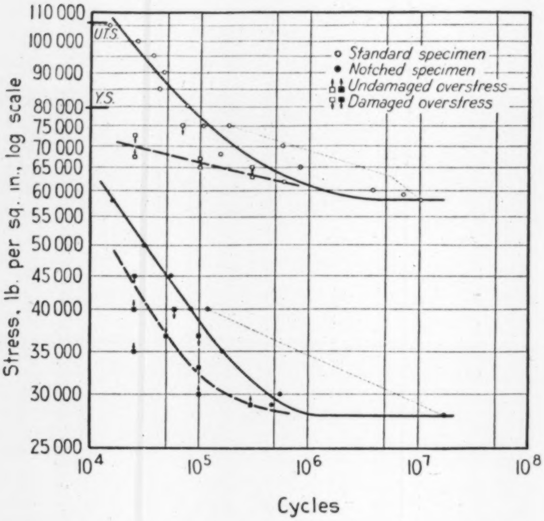

FIG. 8.—Fatigue Curves for Copper Cast Steel Precipitation-Hardened No. 8.
Damage lines are given as heavy dashed lines.

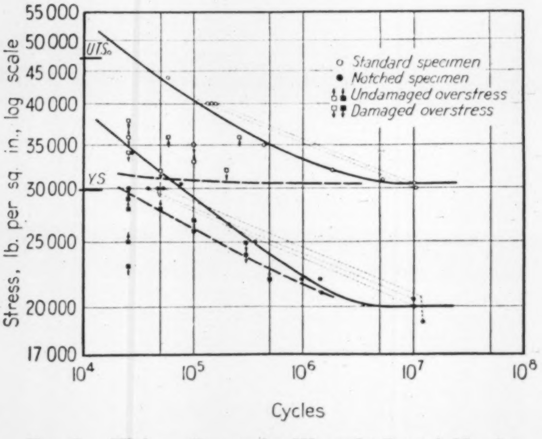

FIG. 9.—Fatigue Curves for Wrought Iron A No. 9.
Damage lines are given as heavy dashed lines.

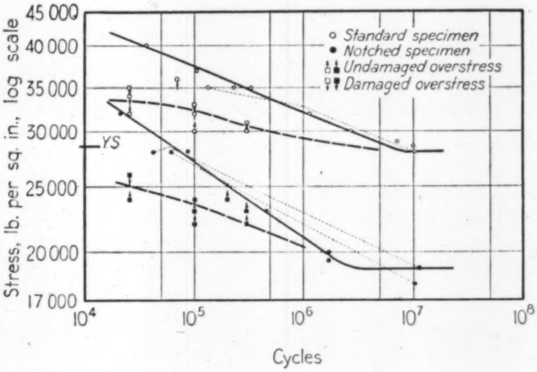

FIG. 10.—Fatigue Curves for Wrought Iron AT No. 10.
Damage lines are given as heavy dashed lines.

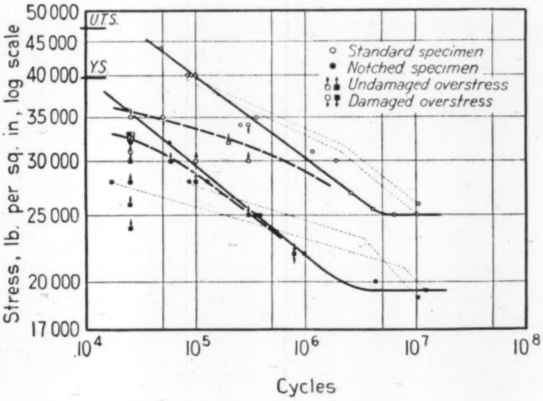

FIG. 11.—Fatigue Curves for Wrought Iron B No. 11.
Damage lines are given as heavy dashed lines.

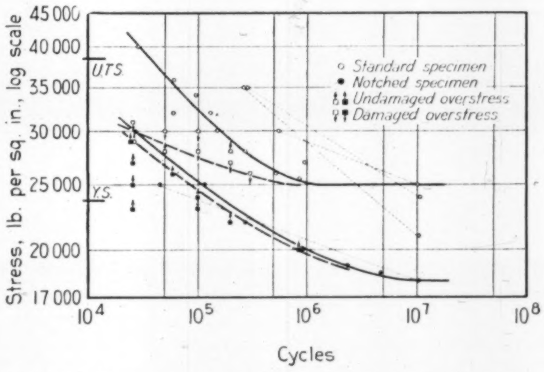

FIG. 12.—Fatigue Curves for Wrought Iron BT No. 12.
Damage lines are given as heavy dashed lines.

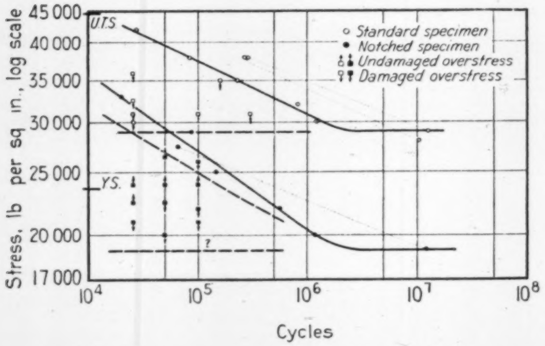

FIG. 13.—Fatigue Curves for Ingot Iron No. 13.
Damage lines are given as heavy dashed lines.

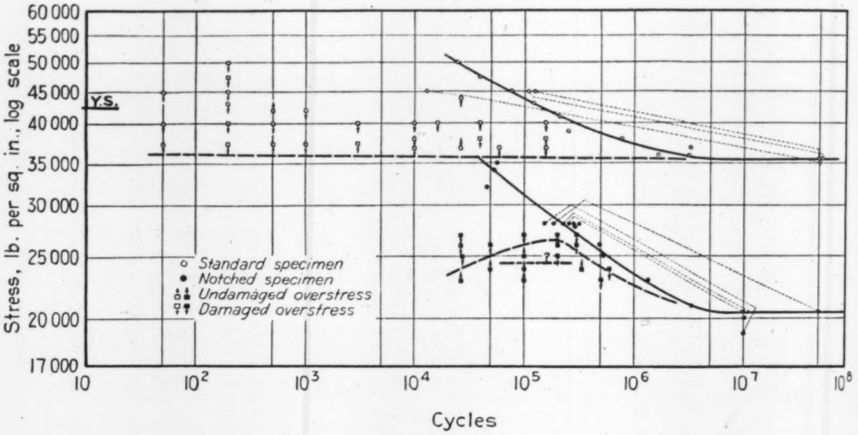

FIG. 14.—Fatigue Curves for 0.35 per cent Carbon Steel No. 14.
Damage lines are given as heavy dashed lines.

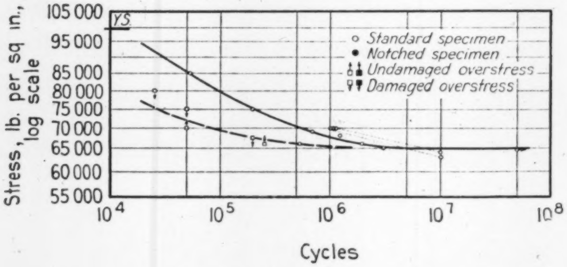

FIG. 15.—Fatigue Curves for S.A.E. No. 4130 Steel No. 15.
Damage lines are given as heavy dashed lines.

load at fracture and the reduced area, was not determined with precision, but is included as one measure of strengthening by cold work. The keyhole and V-notch impact tests are given as a measure of the dynamic ductility and of the response to notches of different sharpness.

The endurance properties are given in Table III. The similarities and differences in the character, as distinct from the magnitude, of the fatigue properties of different materials are best brought out by ratios of stresses. The fatigue ratio, the ratio of the fatigue limit to the tensile strength is a common use of this principle. The relative notch resistance taken as the ratio of the notched fatigue limit to the standard endurance limit has also been used. Its reciprocal is the "stress-concentration factor." In overstress the stress relative to the fatigue limit (unnotched or notched) of similar specimens provides a basis of comparison. Stresses on the stress-cycle curve and the damage line, at 25,000 cycles, have been reduced to relative stresses by dividing by the fatigue limit, and are shown in Table III.

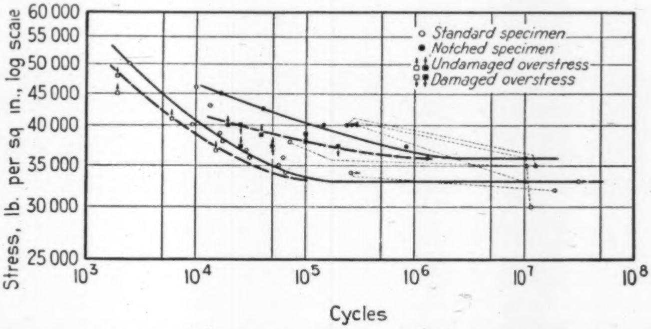

FIG. 16.—Fatigue Curves for 18 per cent Chromium, 8 per cent Nickel Stainless Steel No. 16. Damage lines are given as heavy dashed lines. Note that the curve for notched specimens is above that for standard specimens.

Figures 1 to 16 show the stress-cycle curves and damage lines. The damage lines are represented by heavy dashed lines.

In these curves the stress has been plotted on a logarithmic scale. This is not done with any thought that the curve is a straight line when so plotted, but to aid comparison of the curves. Ratios between stresses are more clearly represented by logarithmic plotting and the shapes of the curves are put on a comparable basis. Yield strengths, and where possible tensile strengths have been indicated on the curves to give a graphic comparison with these static properties.

Virgin specimens which did not fail after 10,000,000 cycles were retested at a higher stress. Points indicating such tests are joined by a dashed line. (The breaks or angles in some of these lines have no significance.) Thus some information on the capacity of the material to be strengthened by understressing is given, but no systematic study was made.

DISCUSSION OF RESULTS

The "expected" endurance limit of a ferrous material is one-half the tensile strength. A material giving an endurance ratio much greater than 0.50 is better than expected and has in a sense unusual resistance to failure by fatigue. Ingot irons and the wrought irons are outstanding with endurance ratios as high as 0.67. Material No. 16 gave the unusually low ratio of 0.38.

In the notched fatigue test the ingot iron and wrought irons again show a high ratio of fatigue limit to tensile strength. Material No. 3 is outstanding with a ratio of 0.50.

In notch resistance the cast steels Nos. 7 and 8 and the 0.35 per cent carbon wrought steel No. 14 show the lowest ratios and hence are the most sensitive to notches. The cast irons and wrought irons Nos. 11 and 12 are moderately insensitive to notches; this is probably one reason why these materials frequently give better performance under repeated stress in service than would be expected from the endurance limit.

With materials having tensile strength from 37,500 to 106,300 lb. per sq. in. (and ignoring materials Nos. 15 and 16) the notched fatigue limit varies only from 18,000 to 31,000 lb. per sq. in. This highest value is given by the crankshaft material No. 3.

The notched fatigue limit of material No. 16 presents a most unusual case, being higher than the endurance limit. The strength of this material is greatly increased by cold working and apparently the root of the notch was unavoidably cold worked in machining.

It has been noted that in general materials with low tensile strengths are least damaged by the presence of notches. Often materials with low strength are ductile, and work harden readily. However, the insensitivity of cast iron to notches cannot be explained in this manner. Cast iron can be considered as an intrinsically strong material weakened by the presence of graphite which serves to produce internal notches or "stress raisers." A deliberately produced notch cannot seriously affect the already notched material. The results with the cast crankshaft material No. 3 which contained only a small amount of graphite, and that (as shown by microscopic examination) mostly in the nodular form, indicate, as might be expected, that a few internal notches are as effective as many.

The high notched-fatigue ratio, and the relatively high ratio of the notched fatigue limit and the endurance limit of the wrought iron may be due not only to the ductility of this material but also to the presence of the slag fibers. Some consistent relation between the notched tests on the longitudinal and transverse specimens of the two lots of wrought iron is to be expected but was not found.

The great ductility and capacity for work hardening of stainless steel would indicate insensitivity to notches, but as previously mentioned some peculiarity resulting from the machining of the specimen seems necessary to explain a notched fatigue limit greater than the endurance limit.

That a damage line exists or, more particularly, that certain specimens could be subjected to overstress without damage, is abundantly evident from the curves. In some cases, due to the moderate number of specimens tested, and to scatter, the position of the damage lines were not located with a very high degree of certainty. Previous knowledge of the 0.35 per cent carbon steel No. 14 showed it to be uniform and the conventional endurance limit showed it to be remarkably free from scatter. Such a material should be ideal for a precise location of the damage line. Unfortunately this particular material showed no resistance to damage by overstress.

The damage line is somewhat similar in shape to the *S-N* curve of the virgin specimens and probably joins it at the endurance limit. The damage line for notched 0.35 per cent carbon steel No. 14 apparently has a maximum stress at 200,000 cycles. Other materials (No. 2 notched and No. 6 standard) gave inconclusive evidence of a maximum in the region of 100,000 cycles. A small overstress for about 25,000 cycles might produce damage while higher overstress for the same number of cycles did not, as shown particularly in the notched ingot iron No. 9.

In discussing the resistance to overstress continued to failure, as indicated by the stress-cycle curve and the resistance to damage by temporary overstress as indicated by the damage line, some numerical index of the position of these curves is desirable. One such index is the ratio of the stress on these curves at 25,000 cycles to the stress at a life of 10,000,000 cycles (the fatigue limit).

Consider first the stress-cycle curve. Unnotched specimens of wrought iron No. 11 and the precipitation-hardened copper cast steel No. 8 withstood stresses 80 and 73 per cent of the endurance limit for 25,000 cycles. Notched, these materials still give high values, and the precipitation-hardened copper malleable No. 6 had the highest with a ratio of 1.90. With the exception of cast iron Nos. 1 and 2 and stainless steel No. 16 the notched specimens withstood higher overstresses relative to their fatigue limits than the unnotched specimens. The curve for notched stainless steel shows the least resistance to continued overstress of any of the materials, notched or unnotched.

The relative resistance to temporary overstress is indicated by the stress on the damage line at 25,000 cycles divided by the fatigue limit. The cast iron No. 1 withstood a 0.50 per cent overstress for 25,000 cycles without damage. Stainless steel No. 16 and wrought iron No. 11 were also resistant to damage. Ingot iron No. 13 and 0.35 per cent carbon steel No. 14 gave no clear indication of resistance to damage, and it is probable that this is characteristic of this class of material. However, molybdenum cast iron No. 2, copper malleable No. 5 and wrought iron No. 9 had no resistance to damage, while other samples in these classes were resistant.

Notched specimens in general were relatively more resistant to damage than the unnotched specimens. A stress 0.75 per cent greater than the notched endurance limit for 25,000 cycles did not damage precipitation-hardened copper malleable No. 6 and many materials withstood 0.50 per cent overstress. All of the wrought irons showed good resistance to damage in the notched condition, while the cast irons all had negligible resistance to damage. Here, as in several other properties, the Ford crankshaft material is in line with the cast irons rather than with the malleable. In spite of, or possibly because of, the high notched fatigue limit of the stainless steel No. 16 it was readily damaged by overstress. The malleables Nos. 4, 5 and 6 showed resistance to damage by overstress, except possibly for material No. 5 for which the data are not conclusive.

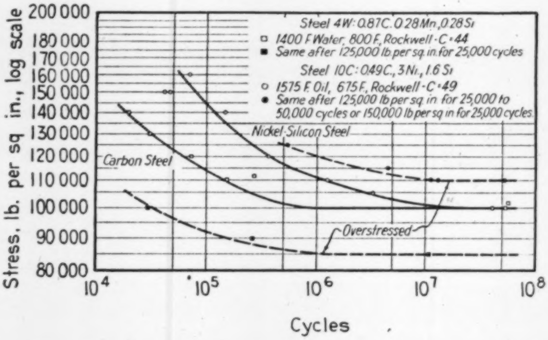

FIG. 17.—Effect of Overstressing on the Fatigue Resistance of 0.87 per cent Carbon Steel and a Nickel-Silicon Steel.

The dashed lines are fatigue curves determined after overstressing. (Taken from H. J. French, *Transactions*, Am. Soc. Steel Treating, Vol. 21, p. 928 (1933).)

It will be noted that precipitation hardening increased the standard fatigue limit of both the copper malleable materials Nos. 5 and 6, and the copper cast steels Nos. 7 and 8, but did not increase the notched fatigue limit. A remarkable effect of improvement in the damage resistance after the precipitation-hardening treatment occurred. This raises the question whether the Ford crankshaft alloy could be treated so as to produce similar improvement.

The effect of overstressing on martensitic steels is discussed at length by French.[3] He showed that steels with tensile strengths approximately 300,000 lb. per sq. in. and fatigue limits as high as 120,000 lb. per sq. in. have varying degrees of resistance to damage by overstress. Figure 17, taken from French's paper is especially interesting in showing two steels with the same endurance limit, one of which (steel No. 4W) had a lower fatigue limit after overstressing, while the other (steel No. 10C) clearly had a higher limit after overstressing.

CONCLUSIONS

Few clear-cut relations between the static properties and the fatigue properties are evident. The conventional endurance limit is, of course, approximately 50 per cent of the tensile strength, but no other relations are even remotely as good. The resistance to notches, as indicated by the ratio of the notched fatigue limit to the unnotched fatigue limit shows a tendency to increase with the ductility, as indicated by the elongation, reduction of area or the impact strength. The cast irons are conspicuous exceptions, and fit better into the statement that there is a tendency for the ratio to increase with decreasing tensile strength. To this the stainless steel No. 16 is a glaring exception, while the crankshaft No. 3 material is completely anomalous.

The resistance to overstress as indicated by the relative stress on the stress-cycle curve at 25,000 cycles and on the damage line seems to be entirely unrelated to the static properties. Tests with a large number of materials, with less difference in structure and composition, would be expected to reveal some tendencies, but apparently a given relation cannot apply to all ferrous materials.

Between the various fatigue tests, correlations are difficult to find. The fatigue limits provide no indication of whether the resistance to continued or intermittent overstress will be high or low. There is an indication that high resistance to continued overstress is associated with high resistance to damage by intermittent stress. This, however, is not a very profound observation, for obviously a low damage line must exist when the stress-cycle curve is low.

It is still too early to judge of the importance of overstress testing or even the best manner of making the tests. Through experience the belief has arisen that certain materials are peculiarly suited to service involving repeated stress. In some cases, notably that of wrought iron, conventional fatigue tests have failed to establish a basis for this belief. Even the knowledge that wrought iron was relatively insensitive to notches failed to justify completely a belief in its suitability. The added fact that wrought iron is unusually resistant to overstress goes far to clear up the question. Similarly the pearlitic malleable No. 3 can be seen to have fatigue properties which explain its success as an automobile crankshaft material.

Where repeated stress is met in practice, it is obvious that if a material is used whose fatigue limit is higher than any stress to which it will be subjected, it should not fail. However, in general, stresses greater than the fatigue limit are inevitable. The fatigue limit is no clear guide to the resistance to overstress and hence to the choice of material. It is believed that the overstress tests here described, will, to a considerable degree, provide a suitable basis of choice.

Speculations on the Mechanism of Fatigue

Fatigue phenomena clearly show evidence that at least two distinct processes are at work, a strengthening process and a damaging process. Both processes become more active with increased stress, though the damaging process may be entirely absent below the endurance limit. The strengthening process appears to be a form of strain hardening. The damaging process is of an imperfectly known nature, but evidently consists of two stages, crack formation and crack propagation. When the crack has become large enough to be seen without the use of refined methods the fracture usually follows immediately. Thus, in the latter stages of fatigue, crack or notch propagation is very rapid. Apparently, however, an incipient crack may be propagated rapidly or slowly, depending upon conditions.

The existence of a damage line argues strongly for the belief that there may be an induction period during which forces tending to form a crack are in operation, but the crack itself does not start. If a specimen which has been overstressed for a time insufficient to start an incipient crack has the stress lowered to a value insufficient to cause crack formation, the specimen will run indefinitely, and the specimen may be said to have not been damaged by the overstress. Because of the probable action of the strengthening process during the initial overstressing, and of the possibility that the notch formation process may act at the fatigue limit stress, it is not safe to conclude that the damage line marks the end of the crack formation process and the beginning of the crack propagation zone.

If we suppose that the damage line represents the beginning of crack propagation then the number of cycles between it and the stress-cycle curve represents the rate of notch propagation. While the plotting of the number of cycles on a logarithmic scale tends to obscure the fact, in general the number of cycles between the damage line and the stress-cycle curve decreases with increased overstress. Thus, as expected, the rate of notch propagation increases with stress. In the few cases where the stress-cycle curve for notched specimens has been carried up to stresses where the damage line of unnotched material is known there is no evident relation between the life of the notched specimen and the number of cycles between the damage line and the stress-cycle curve at the same stress. However there is no reason to believe that the square notch used acts the same as the crack formed on damaging by overstress.

It would be far more satisfactory if some broad generalization could be reached as to just why different ferrous materials vary in ability to stand overload without damage so that one might develop new ones with superior ability in that line. Obviously that has not been accomplished in the present work. Nevertheless there is some degree of concordance between the damage-resisting ability of some of the materials studied and the esteem in which they are held on the basis of performance in service. At any rate, ferrous materials do differ in overstress resistance under the conditions of our tests. That these particular tests are adequate and satisfactory to determine the ability of materials to stand up in actual service is quite unlikely, but we believe they do justify the recommendation that design engineers, testing engineers and metallurgists pay more attention to the conditions of occasional overstress in service, and to the development of alloys suited to endure such stresses without rapid damage.

DISCUSSION

MESSRS. H. F. MOORE[1] AND H. B. WISHART[1] (*presented in written form*).—Messrs. Russell and Welcker have given us a valuable addition to the test data on the effect of occasional stress above the endurance limit, using the French "damage line" method. Their use of an arbitrary standard for damage—the ratio of the stress which causes damage at 25,000 cycles of stress to the virgin endurance limit of the metal—is worthy of careful consideration and furnishes what is at least a tentative yardstick for measuring damage by overstress.

During the past year there have been carried on in connection with the work of the Fatigue of Metals Laboratory at the University of Illinois two series of tests using the damage line method. One series was a study of damage at low temperature carried on by members of the Illinois staff in the cold room at Wright Field, Dayton, Ohio. The results of one series of those tests are given in the accompanying Table I, and in Fig. 1; the remainder are now being reduced and tabulated. The results given are from tests on specimens of 0.75 per cent carbon steel cut from the head of a railroad rail.

TABLE I.—RESULTS OF TESTS ON WROUGHT IRON AND STEEL.

Material	Fatigue Limit, lb. per sq. in.	Fatigue Ratio, Fatigue Limit to Tensile Strength	Relative Stress at 25,000 cycles		Ratio *A*:*B*
			Ratio to Fatigue Limit (*A*)	Ratio to Damage Stress (*B*)	
Engine-bolt wrought iron:					
longitudinal, 70 F	27 000	0.56	1.50	1.05	1.43
transverse, 70 F	21 000	0.68	1.57	1.27	1.24
Structural steel:					
longitudinal, 70 F	30 000	0.51	1.33	1.17	1.17
transverse, 70 F	30 500	0.49	1.41	1.10	1.28
0.75 per cent carbon steel, 70 F	62 000	0.48	1.19	1.06	1.12
10 F	65 000	....	1.29	1.06	1.21
−20 F	71 000	....	1.25	1.05	1.19
−40 F	71 000	....	1.37	1.11	1.23

The second series of tests was carried out in the Fatigue of Metals Laboratory at Illinois as a senior thesis in mechanical engineering by Mr. John W. Luce. He compared the behavior of specimens of engine-bolt wrought iron with that of specimens of structural steel. Both wrought iron specimens and steel specimens were cut from bars 1 by 4 in. in cross-section. The results of these tests are also summarized in the accompanying Table I, which is arranged with the same headings as those used by Messers. Russell and Welcker in their Table III, omitting columns for notched specimens, and adding a column showing the ratios of the values corresponding to those given in the last two columns in the Russell-Welcker Table III.

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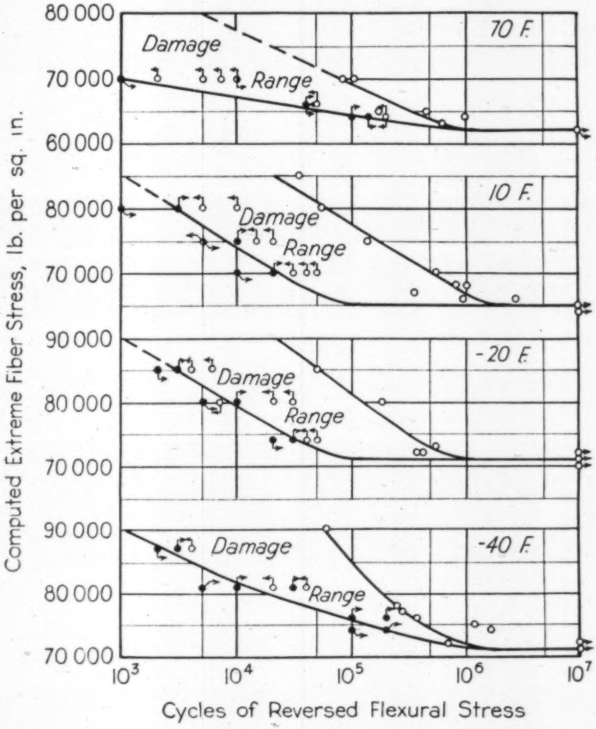

FIG. 1.—Damage Diagram—0.75 per cent Carbon Steel.

These values fall within the range of values found by Russell and Welcker. The low fatigue limit for transverse wrought iron specimens is probably due to large slag streaks. Microscopic examination showed such streaks in the metal.

The speakers have had the same difficulty as that reported by Russell and Welcker, namely, that the slight uncertainty in value of virgin endurance limit causes a magnified degree of uncertainty in locating points on the damage line. A series of tests in which damage line was based, not on the virgin endurance limit, but on some percentage of that limit (say, 95 per cent) might suggest a better technique for getting a measure of damage by overstress. (However, further tests have not borne out this hope.)

The values of the ratio $(A):(B)$, given in the last column of the accompanying Table I indicates, in general, a diminution of the damage area as those values approach unity. At first glance a narrow damage area, indicating that without suffering damage the metal may be repeatedly stressed close up to the point of fracture, seems to be a test result favorable to the metal. In many, perhaps in most, cases this would be true, but for members for which it is possible to examine the member at intervals to see whether there are any signs of fatigue damage (for example, periodic examination of car axles for cracks, or railroad rails for fissures Sperry car test)—for such members a broad damage area might be an indicator of a fatigue crack long before fracture. This would mean that for metals with "broad" damage areas inspections for cracks or other signs of fatigue damage would not have to be made so often as for members made of metal with a "narrow" damage area. A metallographic study of the relation between breadth of damage area and the percentage of "life" at which a fatigue crack can first be detected would seem to be a particularly worth-while line of research.

7

INFLUENCE OF TIME AT 1000 F. ON THE CHARACTERISTICS OF CARBON STEEL

By A. E. White,[1] C. L. Clark[1] and R. L. Wilson[2]

Synopsis

This paper describes extended-time creep tests at 1000 F. on an annealed electric furnace 0.15 per cent carbon steel. The stresses were so selected that rupture of the specimens occurred after different intervals of time in the series of tests, and fractures have actually been obtained in periods up to 14,000 hr. The complete time-elongation curves are thus available, permitting the arrangement of the results to show the influence of time of test on the reported creep stresses.

The investigation reveals that carbon steel loses considerable ductility after prolonged heating under stress at 1000 F. and on the basis of a metallographic examination an explanation is offered for this behavior.

Evidence is also presented to show that (1) the type of fracture varies depending on whether the temperature of test is above or below the lowest temperature of recrystallization, (2) spheroidization will occur in plain carbon steel under stress at 1000 F., and (3) remanent strain-hardening is manifested in this steel at 1000 F. only when the time for rupture is short, as in the short-time tension test.

Introduction

Although many researches have been undertaken on the creep characteristics of metals at elevated temperatures, the greater portion of this work has been confined to stress and temperature conditions such that the resulting deformation or creep has been relatively small. For many commercial applications, and especially those in which the total permissible deformation is slight, tests of this kind yield the desired information for design purposes.

From a theoretical, as well as a practical, standpoint it is believed that valuable information can also be obtained if creep tests are so conducted that a wide range of creep rates is considered. This can be accomplished by so selecting the tensile stresses that actual fracture will result after different periods of time. If the periods required for fracture are sufficiently prolonged, tests of this description will yield information not only with respect to (1) the influence of time on the creep characteristics, but also on (2) the influence of time as well as stress on the ductility exhibited by the fractured specimens, (3) the type of fracture obtained, (4) the stability of the microstructure, and (5) the strain-hardening properties.

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The purpose of this paper is to present the results of creep tests that have been in progress for several thousand hours, in which the stresses were purposely chosen of sufficient magnitude to cause the fracture of S.A.E. No. 1015 plain carbon steel in time periods up to 14,000 hr.

PROCEDURE

The steel for this investigation was commercial electric furnace S.A.E. No. 1015 plain carbon steel obtained from the Timken Steel and Tube Co. The composition is as follows:

Carbon, per cent	Manganese, per cent	Silicon, per cent	Phosphorus, per cent	Sulfur, per cent
0.15	0.50	0.23	0.019	0.021

Specimens were machined from 1-in. round bars heat treated by annealing from 1550 F. to a Brinell hardness of 134. The material was relatively coarse-grained, having an inherent grain size of 4 to 5.

The creep tests were conducted in accordance with the procedure proposed in the A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 35 T).[3] The standard creep-testing apparatus of the University of Michigan, which has been described previously,[4] was used for the tests and all the details of testing were in agreement with those described in the paper before this Society last year.[5]

For the creep tests five stresses ranging from 4000 to 12,000 lb. per sq. in. were applied. From the resulting elongation curves the creep rates were determined at approximately 500-hr. intervals and, by plotting these rates against time, the influence of time on the creep rate was obtained. The creep rates for the various times were then plotted on logarithmic coordinates against the corresponding unit stresses and from these curves the stress values for the definite creep rates were obtained. These values were then plotted against time to indicate the influence of time on the creep characteristics.

The ductility of the steel was determined through measurements of the gage section and the diameter. An explanation for the observed decrease in the ductility was obtained by subjecting the surface of the specimens to metallographic examination. The actual fractures were also studied microscopically in order to determine the path of the rupture, and to observe the stability of the structure near the fracture. The strain hardening of the steel, for the test conditions given, was estimated from Vickers hardness readings taken over the $\frac{3}{4}$-in. section adjacent to the fracture.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1291 (1935); also 1935 Book of A.S.T.M. Tentative Standards, p. 1415.

[4] A. E. White, C. L. Clark and L. Thomassen, "An Apparatus for the Determination of Creep at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs. Vol. 52, No. 27, p. 347 (1930).

[5] A. E. White, C. L. Clark and R. L. Wilson, "Influence of Time on Creep of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 167 (1935).

EXPERIMENTAL RESULTS

The results of the tests have been arranged to show the influence of time at 1000 F. on the creep properties, the ductility of the fractured specimens, the type of fracture obtained, the stability of the structure, and the strain-hardening characteristics.

Influence of Time on the Creep Characteristics:

This phase of the investigation is a continuation of the work reported before the Society last year.[5] The tests have been continued on the specimens subjected to unit stresses of 4000 and 6000 lb., and three additional tests were conducted using stresses of 9000, 10,000, and 12,000 lb., respectively. All these specimens, with the exception of the one under the 4000-lb. stress, have already fractured after time periods varying from 1552 to 13,950 hr.

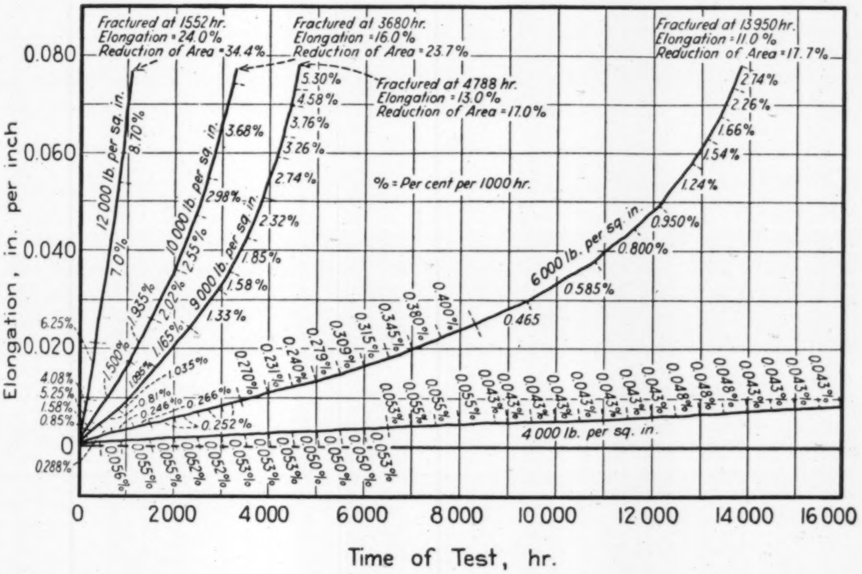

FIG. 1.—Time-Elongation Curves at 1000 F. for Annealed Electric-Furnace Carbon Steel.

The time-elongation curves setting forth the results obtained are given in Fig. 1. Figures 2 and 3 show the influence of time on the reported creep rate, and Fig. 4 the influence of time on the creep strength. In all cases the creep rates at the various designated times are the rates which would have been reported had the tests been discontinued at that particular time. These rates of creep were determined over the entire duration of the test and thus the first and third stages of creep, as well as the second, are included. For the second stage of creep, time periods of 500 hr. were used in determining the creep rates while in the other two stages the periods were

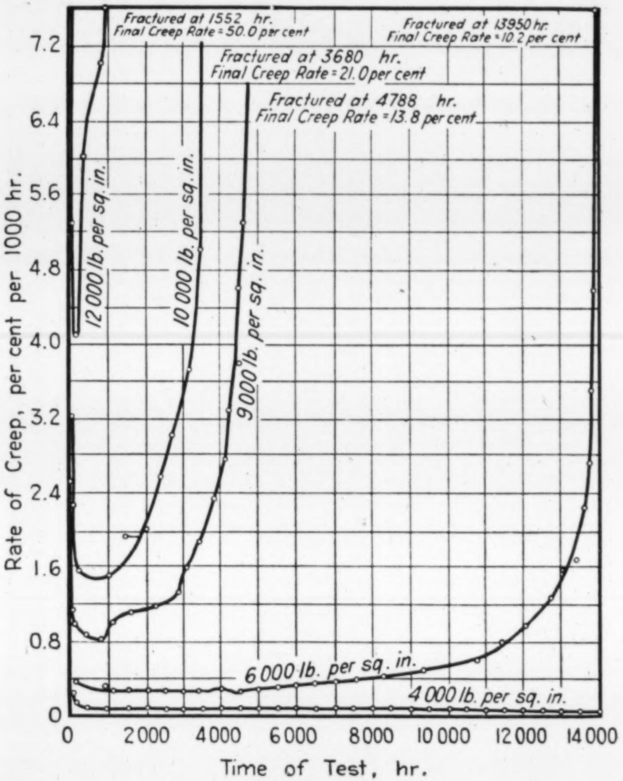

FIG. 2.—Influence of Time of Test on the Observed Creep Rate of Annealed Electric-Furnace Carbon Steel at 1000 F.

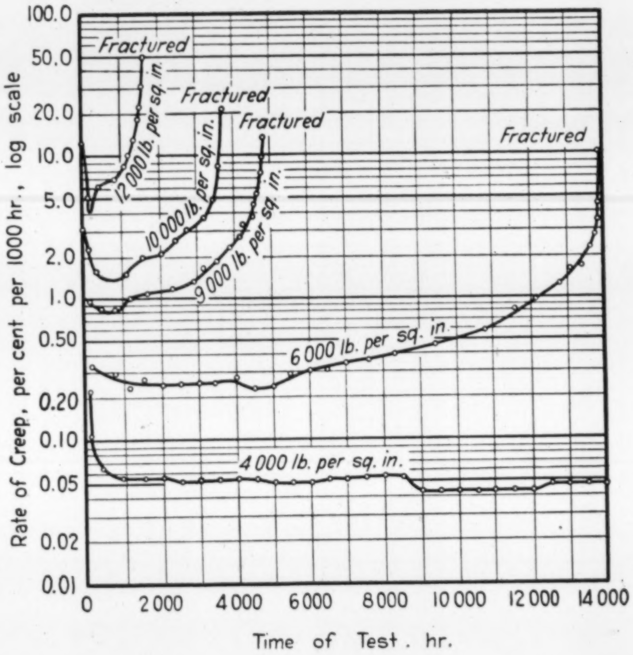

FIG. 3.—Influence of Time of Test on the Observed Creep Rate of Annealed Electric-Furnace Carbon Steel at 1000 F. (Semi-Logarithmic Coordinates).

generally much shorter because of the rapidly decreasing creep rate in the first stage and increasing creep rate in the third stage. The creep rates expressed in per cent per 1000 hr. are indicated on the time-elongation curves of Fig. 1.

All the specimens that fractured exhibited three stages of creep as would be expected. The following table shows the effect of stress on the length of time the specimens were in the various stages of creep:

	Duration of Various Stages of Creep, hr.		
Stress, lb. per sq. in.	First Stage	Second Stage	Third Stage
4 000	1000	13 000[a]	....
6 000	1500	3 500	9000
9 000	400	400	3988
10 000	250	750	2680
12 000	100	50	1402

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[a] Test still in progress.

From these values it appears that the duration of each stage of creep tends to decrease with increasing stress.

This table also demonstrates that fracture will not necessarily occur soon after the third stage of creep is entered. The length of the third stage varied inversely with the stress and for the 6000-lb. stress it lasted 9000 hr. (for this same stress the second stage of creep was only of 3500 hr. duration). The total deformation occurring in the third stage, however, varied directly with the stress; that is, the greater the stress the greater the deformation in this stage. If appreciable oxidation occurs, this will likewise influence both the second and third stage characteristics.

Figures 2 and 3 showing the influence of time on the reported creep rate differ only in that Fig. 3 is plotted to semi-logarithmic coordinates. With all the stresses employed the creep rate first decreased, then remained steady for a certain period of time, and finally, if the stress was sufficiently great, the creep rate increased until fracture occurred. It is interesting to note that, for this particular steel at least, the magnitude of the final creep rate before fracture varied directly with the stress. This variation, however, is not linear—whether plotted cartesian, semi-log, or log-log.

The creep rates for the various time periods were plotted against the corresponding stresses on logarithmic coordinates to obtain the stresses that would produce creep at the rate of 0.01, 0.10, and 1.0 per cent per 1000 hr. These creep stresses were then plotted against time as in Fig. 4.

Figure 4 shows that the stresses required for creep rates of 0.01 and 0.10 per cent per 1000 hr. remained practically constant for the first 6000 hr. Thereafter the stress for the lower creep rate increased and for the higher rate decreased. However, the changes are not especially marked. For example, the total range of stress for creep at the rate of 0.01 per cent per 1000 hr. is from 2500 to 3400 lb. per sq. in., while for the 0.10 per cent rate it is from 4300 to 4800 lb. per sq. in. The maximum variations are therefore 900 and 500 lb. per sq. in., respectively.

In the case of the stress required for creep at the rate of 1.0 per cent per 1000 hr., time had a more pronounced influence. The stress decreased continuously as the testing time was increased and the values ranged from 9000 down to 5400 lb. per sq. in. This represents a difference of 3600 lb. per sq. in., which is many times greater than was found for the two lower creep rates.

It should be noted that the curves of Fig. 4 tend to converge as the testing time is extended. The maximum difference between these curves exists at 500 hr. and amounts to 6500 lb. per sq. in.; the minimum spread of 2000 lb. per sq. in. occurs at 14,000 hr. Evidently the difference between the strength values of the steel for the several rates of creep becomes smaller as the time of testing is increased. Perhaps these curves might actually come together if the tests were run long enough.

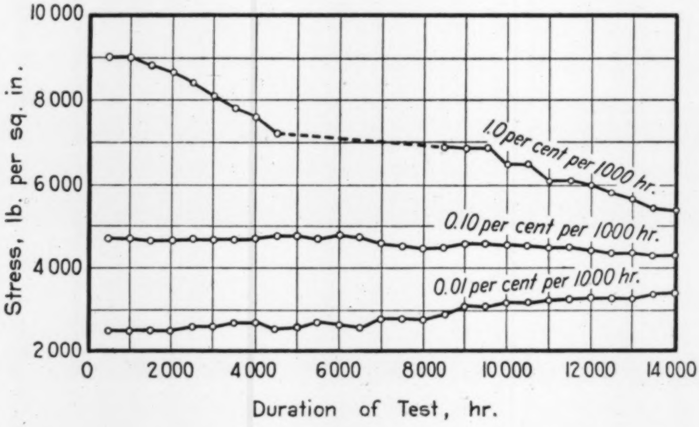


FIG. 4.—Influence of Duration of Test on Reported Creep Stress of Carbon Steel at 1000 F.

This condition is at first somewhat difficult to conceive as it implies that, in tests conducted for greatly extended time periods, the creep rate is governed by the temperature and the character of the steel and is independent of the applied stress. It would mean, for instance, that for this particular steel at 1000 F. there is only one stress under which creep will continue indefinitely. With higher stress fracture will take place ultimately, while with lower stress perceptible creep would stop.

Convergence of the creep-strength curves for the various rates of creep is due largely to the manner in which the curves have been constructed. By including data for high stresses and fast creep rates, such as would surely cause rupture in a reasonable time, the interpolated values are shifted higher for slow rates of creep and lower for fast rates of creep.

While it is well to recognize this shortcoming in the customary method of determining creep strength the practice should not be criticized too severely as there would be few comparative creep data available if all the tests were simply a record of unrelated load-extension curves for materials under low stresses.

Ductility versus Time for Fracture:

The ductility, or the amount of elongation and reduction of area that a piece of steel undergoes before fracture, is regarded as one of the important engineering properties of materials. If the ductility is great, the material will stretch considerably before rupture and thus give warning of impending failure. This is of particular value in connection with high-temperature apparatus where plastic flow of metal takes place easily on slight overstress. To know the influence of time and stress on the ductile properties of a steel at high temperatures may therefore be useful in selecting materials of construction for certain applications.

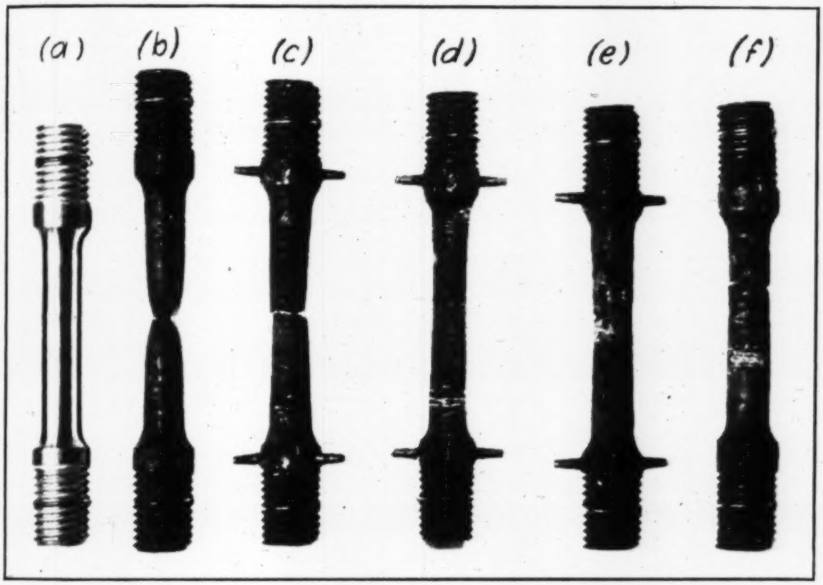


FIG. 5.—Influence of Time on the Resulting Fracture of Annealed Carbon Steel at 1000 F.

(a) Original Specimen.

(b) Tensile Specimen. 1000 F., Tensile strength, 36,500 lb. per sq. in.; elongation in 2 in., 42.5 per cent; reduction of area, 76.9 per cent.

(c) Creep Specimen. 1000 F.; 12,000 lb. per sq. in.; 1552 hr. for rupture; elongation in 2 in., 24.0 per cent; reduction of area, 34.4 per cent.

(d) Creep Specimen. 1000 F.; 10,000 lb. per sq. in.; 3680 hr. for rupture; elongation in 2 in., 16.0 per cent; reduction of area, 21.3 per cent.

(e) Creep Specimen. 1000 F.; 9000 lb. per sq. in., 4788 hr. for rupture; elongation in 2 in., 13.0 per cent; reduction of area, 18.4 per cent.

(f) Creep Specimen. 1000 F., 6000 lb per sq. in., 13,950 hr. for rupture; elongation in 2 in., 11.0 per cent; reduction of area, 17.7 per cent.

In the extended-time creep tests, all of the specimens except the one under the lowest stress have fractured. As the specimens ruptured after different periods of time depending upon the load applied, it was possible to measure the ductile properties of the carbon steel under several conditions of time and stress for comparison. Figure 5 shows the fractured specimens. The elongation in 2 in. and reduction of area are indicated for each specimen and are also shown graphically in Fig. 6.

From these values it is evident that the ductile properties of this steel are greatly decreased as the time for fracture is increased. For example, the short-time tension specimen gave an elongation of 42.5 per cent and a reduction of area of 76.9 per cent. The corresponding values for the creep specimen which required 13,950 hr. for fracture are only 11.0 and 17.7 per cent. The ductility values for the remaining fractured specimens are decidedly below those of the short-time tension specimens with the maximum decrease in the ductility occurring during the first 5000 hr.

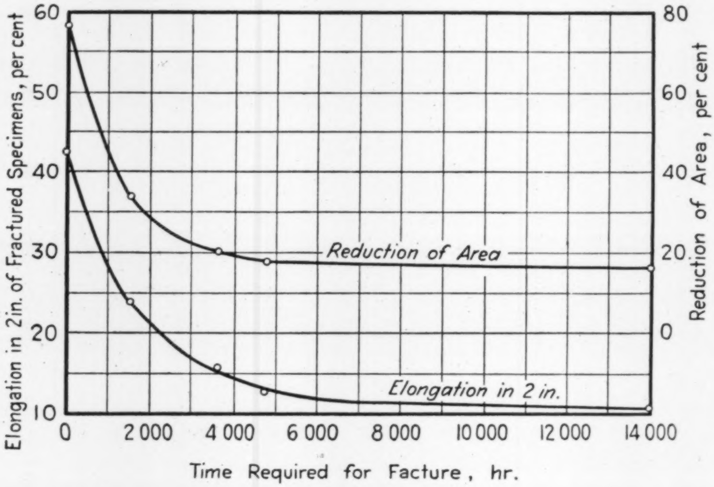


FIG. 6.—Influence of Time Required for Rupture at 1000 F. on the Ductility of S. A. E. No. 1015 Carbon Steel.

Referring to the results previously presented for the chromium-silicon-molybdenum steel[5] at this same temperature, it will be found that a similar decrease in ductility was not observed:

STRESS	TIME FOR RUPTURE, HR.	ELONGATION IN 2 IN., PER CENT	REDUCTION OF AREA, PER CENT
Short-time tension specimen	....	32.5	78.6
34 000	2625	25.0	70.3
30 000	3250	21.0	67.9
24 600	6151	28.5	72.8

None of the fractured creep specimens possessed as high ductility as the short-time tension specimens but the amount of decrease was not so pronounced as in the case of carbon steel. Furthermore, the ductility of the alloy steel did not decrease progressively as the testing time was increased.

The surface of the creep specimens was examined metallographically in order to determine why the carbon steel showed this marked decrease in ductility while the chromium-silicon-molybdenum steel did not. Loss of ductile properties is apparently due to disintegration of the carbon steel caused by formation of oxide between the grains at the surface of the specimens. The extreme conditions of the structures with respect to intergranular oxidation are shown in the photomicrographs of Fig. 7.

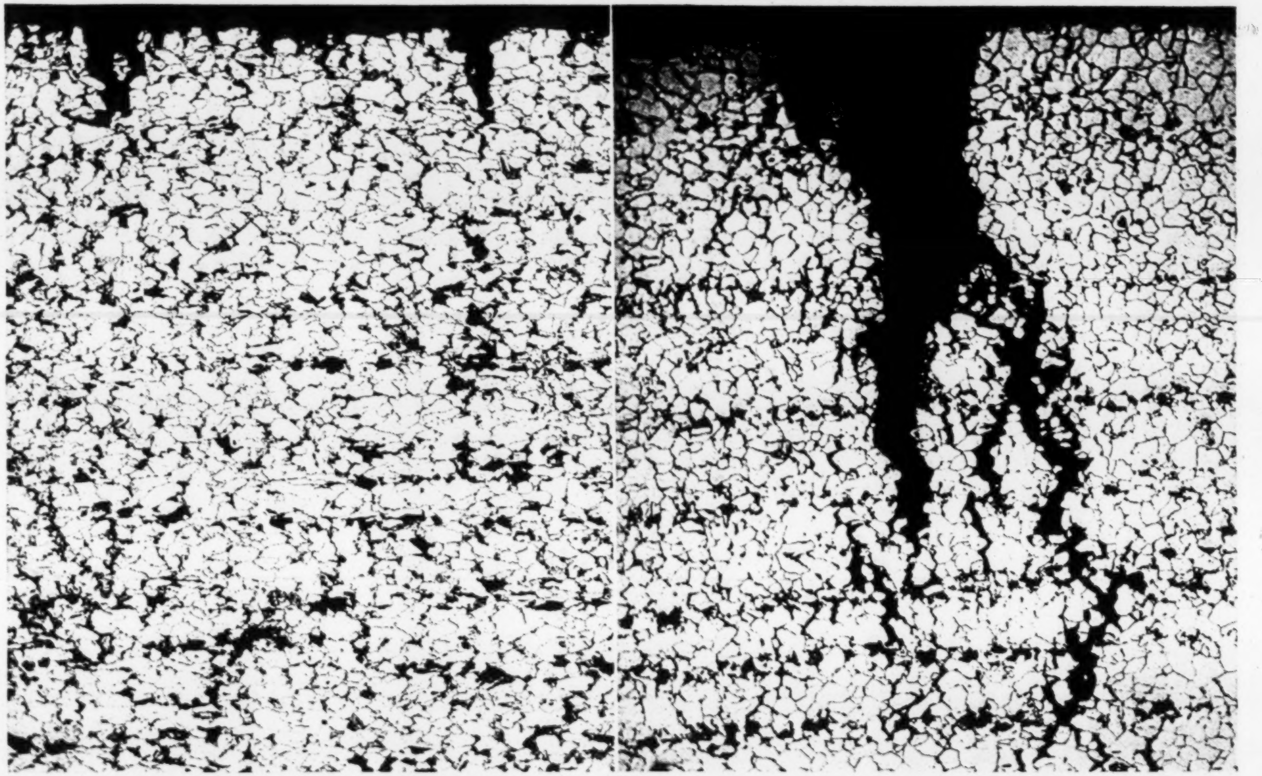
While the atmosphere in the creep-test furnaces is oxidizing, air cannot circulate freely through the furnaces as the top is entirely closed and the bottom packed with asbestos. In fact, the general oxidation of the carbon steel creep specimens was not appreciable. However, there was pronounced intercrystalline oxidation, the average depth of penetration being proportional to the duration of the tests. For example, the average depth of intercrystalline oxidation was 0.007 in. for the specimen under 12,000 lb. per sq. in. stress and 0.040 in. for the one under 6000 lb. per sq. in. stress. Formation of intergranular oxide seems to explain not only the low ductility of the steel but also the progressive deterioration of the metal as the testing time was increased. This explanation is strengthened by the fact that the chromium-silicon-molybdenum steel did not show significant loss of ductility and at the same time did not show intergranular oxidation.

From the results of the microscopic examination conducted on the fractured creep specimens of carbon steel, the possible relation between creep strength and oxidation resistance of a steel can be imagined. If a steel is liable to oxidation because of its chemical composition, the parting of grains in the structure due to intergranular oxidation has the effect of reducing the section and increasing the creep for a given applied load. This might warrant the conjecture that alloying elements capable of improving oxidation resistance would increase the creep strength of a steel, although this is not always borne out. It could also be said with equal force that the atmosphere to which the steel is exposed would sometimes have an influence on the creep strength and ductile properties of the material.

Type of Fracture versus Time:

According to one theory which has been advanced, the type of fracture obtained for any given steel should vary depending upon the temperature and the length of time required for fracture. Below the equi-cohesive temperature or lowest temperature of recrystallization the fracture should be transcrystalline regardless of the time required for rupture, while above the equi-cohesive temperature the fracture should be transcrystalline on rapid breaking and intercrystalline when a sufficiently long time is taken for rupture.

The fractured creep specimens in the present investigation fulfilled the conditions of slow loading above the equi-cohesive temperature that are

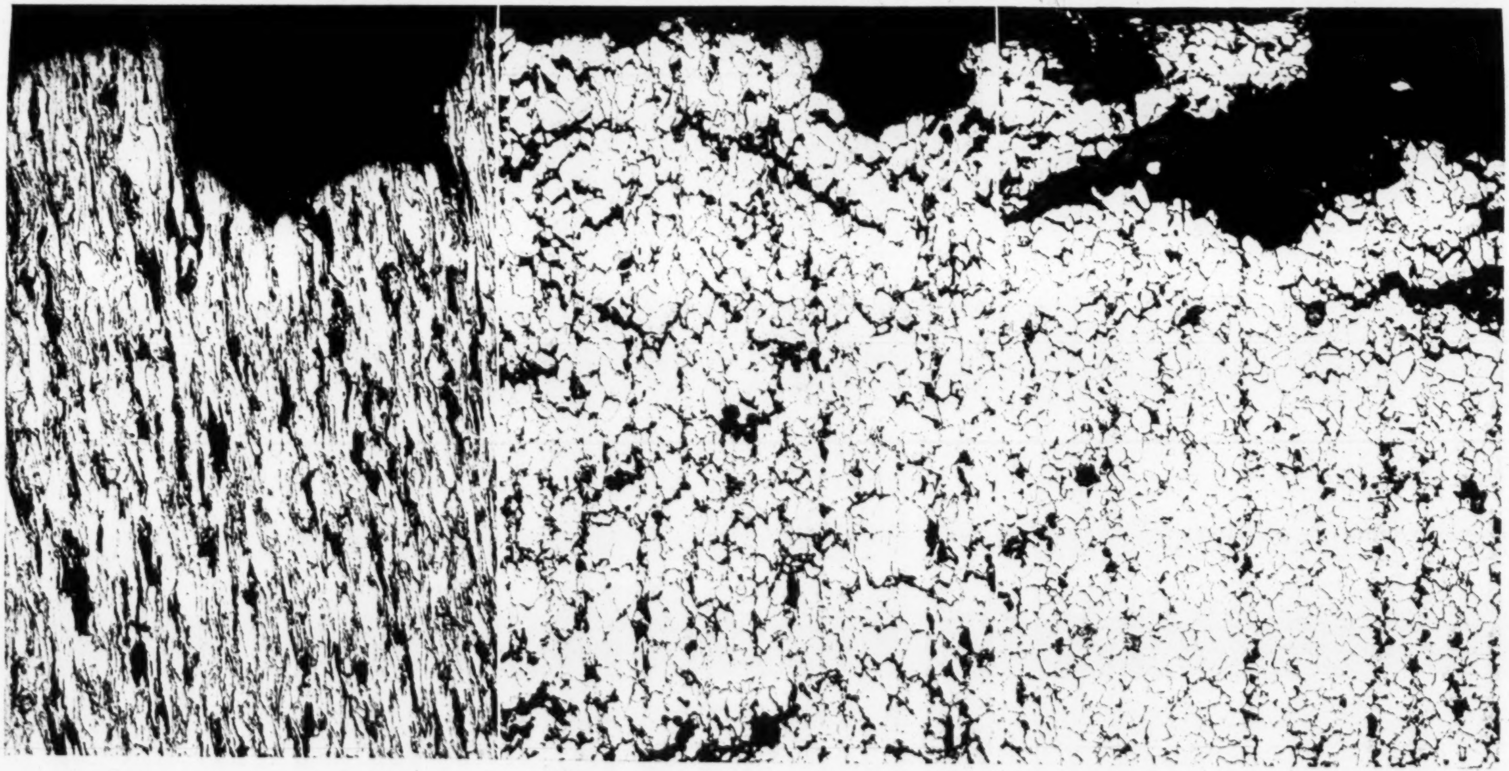


(*a*) 12,000 lb. per sq. in. 1552 hr. for rupture.

(*b*) 6000 lb. per sq. in., 13,950 hr. for rupture.

FIG. 7.—Microstructures of the Surface Sections of Two of the Creep Specimens Showing Intergranular Oxidation (× 100).

FIG. 7.—Microstructures of the Surface Sections of Two of the Creep Specimens Showing Intergranular Oxidation (× 100).



(*a*) Short-time tension specimen. Note strain hardening and transcrystalline fracture.

(*b*) Creep specimen. 12,000 lb. per sq. in., 1552 hr. for rupture. Note lack of strain hardening and intercrystalline fracture.

(*c*) Creep specimen. 6000 lb. per sq. in.; 13,950 hr. for rupture. Note lack of strain hardening and intercrystalline fracture.

FIG. 8.—Microstructures of Short-time Tension and Creep Specimen Fractures Showing Influence of Time on Type of Fracture Obtained (× 100).

supposed to give an intercrystalline fracture. The short-time tension tests might also have been pulled fast enough to give a transcrystalline rupture. A metallographic examination was therefore made on each of the specimens from the creep tests and the short-time tension tests. The conditions found are illustrated by the three photomicrographs of Fig. 8. These photomicrographs represent the typical structures at the fracture of the short-time tension specimens and the two creep specimens which required the shortest and the longest time periods for rupture.

Fracture at 1000 F. in the short-time tension test is largely transcrystalline, Fig. 8 (*a*). Both the ferrite and pearlite grains are distorted and elongated parallel with the axis of the specimen. This indicates that considerable strain-hardening occurred during the test with little if any recrystallization. In other words, under these conditions the rate of strain-hardening greatly exceeded the rate of recrystallization.

All the creep specimens of this steel showed an entirely different type of fracture, as illustrated by Fig. 8 (*b*) and (*c*). The fracture is intercrystalline and the grains, even immediately adjacent to the fracture, are small and equi-axed and are thus free from strain or distortion. If appreciable strain-hardening did occur in these instances, the rate of recrystallization exceeded the rate of strain hardening so that the specimen, upon the completion of the test, was free from strain. It also follows that 1000 F., which is the temperature of the tests, must lie above the equi-cohesive temperature of the plain carbon steel.

On comparing these fractures with those previously presented[5] for the chromium-silicon-molybdenum steel, marked differences will be found to exist. All the fractures of the alloy steel were similar to the one shown for the short-time tension specimen of the carbon steel. That is, the fractures of the alloy steel were largely transcrystalline and the grains in the vicinity of the fractures were distorted. This means that the rate of strain-hardening exceeded the rate of recrystallization and consequently that the test temperature employed, 1000 F., is below the lowest temperature of recrystallization for the alloy analysis.

The results of the tests on both steels therefore substantiate the abovementioned theory concerning the influence of temperature and time on the type of fracture obtained. In rapid breaking, the tension fracture is transcrystalline and the grains in the vicinity of the fracture are distorted, while in slow breaking the type of fracture depends on whether the test temperature is below or above the lowest temperature of recrystallization. If it is below, the fracture will be the same as the type obtained in rapid breaking, while if it is above, the fracture will be intercrystalline and the grains will be free from strain and distortion.

Structural Stability:

Stability of steels is manifested by absence of changes in microstructure resulting from spheroidization of carbide in the pearlite grains. Most of the experimental work on this subject has been confined to tests conducted at temperatures of 1100 to 1300 F. and estimates as to the length of time required for spheroidization at lower temperatures have been based largely on extrapolations. Likewise, while the effect of stress on spheroidization has been recognized as a factor of importance, few test results are available in which this point was considered.

Since the applied loads were constant and the temperature was regulated closely throughout this series of creep tests, an examination of the specimens should indicate to what extent spheroidization has occurred under the various conditions. A comparison of the structures in the shoulder and the gage section of the specimens ought to show the influence of stress. The typical microstructures are shown, at a magnification of 1000 diameters, in Figs. 9 and 10.

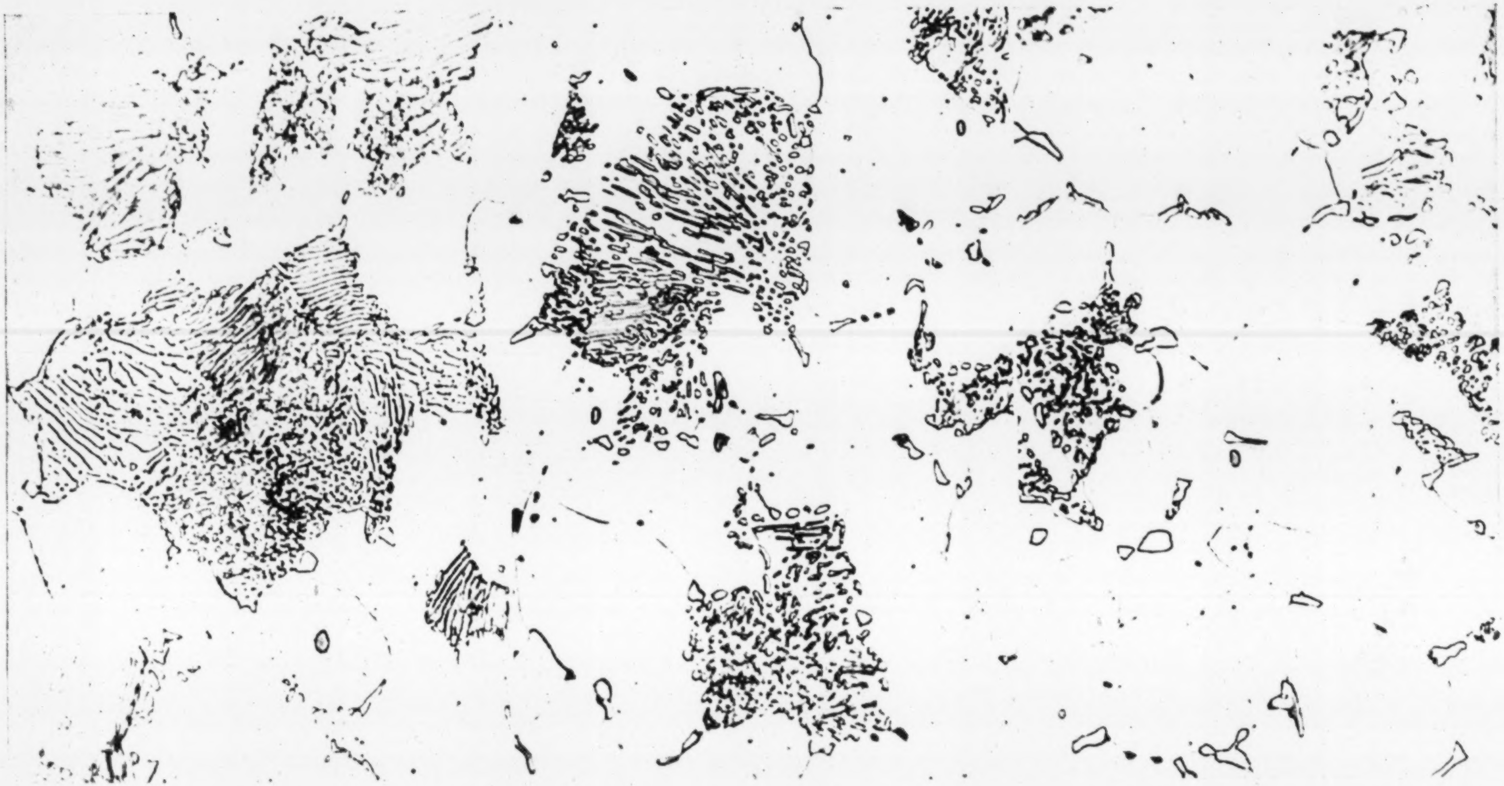
The initial structure of the steel, Fig. 9(*a*), contained pearlite grains that were largely, although not completely, in the lamellar form. The short-time tension tests as well as the creep tests under 12,000 and 10,000 lb. per sq. in. stress produced no apparent change in the pearlite. In the gage section of the creep specimen under 9000 lb. per sq. in. stress (4788 hr. for rupture) spheroidization was practically complete, and in the specimen under 6000 lb. per sq. in. stress (13,950 hr. for rupture) spheroidization was not only complete but considerable migration of the spheroidized carbide particles had occurred. Figure 9(*b*) and (*c*) show the structures of these two creep specimens.

The structures in the shoulders of the specimens which required 4788 and 13,950 hr. for rupture are shown in the two photomicrographs of Fig. 10. By comparing the structures in Fig. 10(*a*) and (*b*) with those in Fig. 9(*b*) and (*c*) the influence of stress on the rate of spheroidization will be apparent. The specimen that broke in 4788 hr. showed spheroidization in the gage section but not in the shoulder; the one requiring 13,950 hr. for rupture showed spheroidization in both locations although it was not nearly as pronounced in the shoulder as in the gage section.

Spheroidization by heat alone at 1000 F. is known to proceed quite slowly. From the results of this investigation it seems that stress acts as a mechanical aid to atomic mobility in the acceleration of carbide movement at elevated temperatures. When the stress is sufficiently great, spheroidization in plain carbon steel at 1000 F. begins to be noticed after about 4000 hr.

Strain-Hardening versus Time:

In order to determine differences in the strain-hardening rates of the short-time tension and creep specimens and thus confirm the conclusions drawn from the metallographic examination of the fractures, hardness tests were conducted on the interior of each of the fractured specimens after splitting them lengthwise over the $\frac{3}{4}$-in. section adjacent to the fracture.

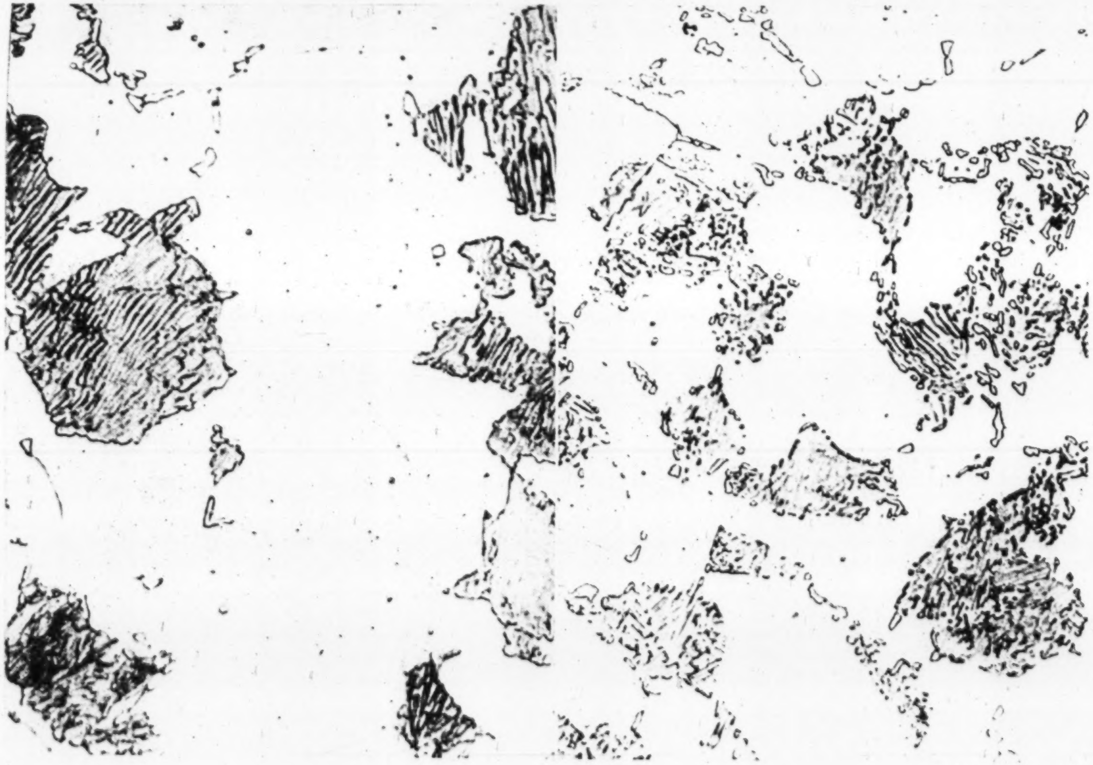


(*a*) Original structure, spheroidized and lamellar pearlite.

(*b*) 9000 lb. per sq. n.; 4788 hr. for rupture. Almost complete spheroidization.

(*c*) 6000 lb. per sq. in., 13,950 hr. for rupture. Spheroidization plus migration.

FIG. 9.—Microstructures Showing Influence of Time and Stress at 1000 F. on Spheroidization (× 1000).



(*a*) Section in shoulder of 9000 lb. per sq. in. specimen. 4000 lb. per sq. in., 4788 hr. for rupture. No spheroidization.

(*b*) Section in shoulder of 6000 lb. per sq. in. specimen. 2670 lb. per sq. in., 13,950 hr. for rupture. Almost complete spheroidization.

FIG. 10.—Microstructures Which Together with Those of Fig. 9 Show Influence of Time and Stress on the Spheroidization of Carbon Steel at 1000 F. (× 1000).

The Vickers test, using a 30-kg. load and a diamond pyramid, was found to be the most suitable. The results obtained are given below:

VICKERS HARDNESS TESTS ON FRACTURED TENSION AND CREEP SPECIMENS.

Position on Specimen	Original	Tension Specimen	Creep Specimens			
			12000 lb. per sq. in.	10000 lb. per sq. in.	9000 lb. per sq. in.	6000 lb. per sq. in.
¾ in. from fracture		136	100	98	94	93
		136	100	98	94	93
	112	136	98	97	94	93
		138	98	95	94	93
		138	92	87	94	86
↓		138	87	87	87	86
At fracture		138	87	87	87	86

It is apparent from these values that a strain-hardening effect was obtained only in the short-time tension specimens. Even in this case, the increase in hardness was not great as the original material showed a Vickers hardness of 112 as compared to an average value of 137 for the short-time tension specimen. All the creep specimens were lower in hardness than the original material, indicating that further softening occurred during the test.

The highest hardness measured on the creep specimens became lower as the length of time for fracture increased. Again the variations in hardness were not especially marked as the greatest range was from 93 to 100, a difference of only 7 points. On the other hand, the hardness immediately adjacent to the fracture reached the same low value in all cases and was thus independent of the time required for rupture. The hardness gradient over this section may be accounted for on the basis that the total deformation varied inversely as the distance from the fracture and thus the amount of recrystallization would vary directly.

These hardness values therefore agree with the results of the metallographic examination as they show that remanent strain-hardening is manifested only in the short-time tension test specimen.

CONCLUSIONS

The results obtained permit the following conclusions regarding the influence of time and stress at 1000 F. on the characteristics of an electric furnace 0.15 per cent carbon steel:

1. The time-elongation curves of creep specimens under stresses of 6000 lb. per sq. in. and greater have all shown three stages of creep. The duration of each stage tends to vary inversely with the stress and is thus proportional to the length of time required for fracture.

2. Both the total deformation during the complete creep test and the

final creep rate are proportional to the stress and thus vary inversely as the time required for rupture. This condition may be dependent on the rate of oxidation as well as the applied stress.

3. The reported creep stresses, being dependent on the creep rates, likewise vary with time. For the plain carbon steel, however, the amount of variation for creep rates of 0.01 and 0.10 per cent per 1000 hr. is not so pronounced as for the chromium-silicon-molybdenum steel studied previously. A larger variation was, however, observed for the 1.0 per cent creep stress as the values ranged from 9000 to 5400 lb.

4. The reported stresses for the creep rates of 0.01 and 0.10 per cent per 1000 hr. remained approximately the same for the first 6000 hr. of the test and were, therefore, independent of the testing time. After this period the stress for the 0.01 per cent creep rate increased while that for the 0.10 per cent rate decreased. On the other hand, the stress for the highest creep rate, 1.0 per cent per 1000 hr., decreased rapidly over the entire testing period.

5. Since the stresses for these three creep rates are approaching one another as the testing time is extended, there is a possibility that, given sufficient time, they will actually reach the same value. This would indicate that the final creep rate at 1000 F., after very extended testing times, is characteristic of the steel and temperature and independent of the applied stress. In other words, there would be only one stress under which continuous creep would occur indefinitely.

6. The assumption ordinarily made that a creep rate of 0.10 per cent per 1000 hr. is equivalent to a creep rate of 1.0 per cent per 10,000 hr. is approximately true for this steel at 1000 F.

7. The 500-hr. test correctly prophesies the creep strengths of the carbon steel at 1000 F. for periods up to 6000 hr. for the 0.01 and 0.10 per cent creep rates. For the highest creep rate considered, however, the shorter tests give no indication of the behavior under more prolonged periods.

8. Given sufficient time, spheroidization will occur in this material under stress at 1000 F. For the test conditions given spheroidization began after about 4000 hr. After 13,950 hr. not only was spheroidization complete but considerable migration of the spheroidized carbide particles had occurred.

9. The fracture of the creep specimens was intercrystalline while that of the short-time tension specimens was transcrystalline. Moreover, the creep specimens showed no evidences of strain-hardening in the vicinity of the fracture while the short-time specimens did. These findings substantiate the theory usually advanced to explain the influence of time and temperature on the type of fracture observed in tension test specimens broken at temperatures above the equi-cohesive temperature.

10. The ductile properties of the fractured creep specimens decreased progressively as the time required for fracture was increased. This was found to be due to the intergranular oxidation which occurred during the tests.

11. Hardness tests showed that a strain-hardening effect was obtained only in the short-time tension specimens, which were broken much more rapidly than the creep specimens. This indicates that the equi-cohesive temperature, or lowest temperature of recrystallization for this material is below 1000 F.

12. These results, together with those previously reported[5] for the chromium-silicon-molybdenum steel, therefore, indicate that the influence of testing time on the high temperature physical properties of steel may not always be the same at a given temperature. The characteristic behavior of the alloy and the plain carbon steel is believed to be due to differences in the lowest temperature of recrystallization. In the case of the plain carbon steel, 1000 F. is above the lowest temperature of recrystallization and the tendency toward recrystallization predominates. On the other hand, for the chromium-silicon-molybdenum steel 1000 F. is either below or within the recrystallization range so that the rate of strain-hardening exceeds the rate of recrystallization.

Acknowledgments:

The authors desire to express their appreciation to The Timken Steel and Tube Co. for the financial assistance which has made these investigations possible and for permission to publish the findings. Particularly do they wish to express their gratitude to F. J. Griffiths, H. H. Timken, Jr., and K. B. Bowman.

They also wish to express their appreciation to J. W. Freeman for assistance in performing the creep tests and for doing the metallographic examination.

DISCUSSION

MR. C. R. AUSTIN[1] (*presented in written form*).—The data furnished by the authors of this paper are extremely interesting and develop information on the effects of unusually prolonged periods of testing.

For a number of years many writers interested in this field of investigation have maintained that satisfactory design data could only be obtained with more prolonged time of testing than is now customarily employed. Others have felt that service tests could be the only true criterion as to the behavior of metals at elevated temperatures and that laboratory creep tests could do no more than indicate the order of magnitude of the working stresses of various alloys after certain prescribed methods of fabrication and heat treatment.

To the writer the present paper appears to lend considerable support to the latter view and to demonstrate that a "creep test" cannot evaluate the ultimate physical and mechanical characteristics of an alloy which is to be placed in service for a period of time far longer than it is economically feasible to conduct the test.

It is noted that the authors have used the same method of plotting creep rate against time of test (Fig. 2) as was used in their paper presented before this Society a year ago.[2] The peculiar jogs in the curves, if real, are difficult to understand. These irregularities, however, give rise to the irregularities found in the derived curves plotted in Fig. 4 (also in Fig. 9 of the authors' earlier paper). The deduction arrived at by the authors from these curves is interesting and appears to warrant no apology, and it would appear that a similar conclusion could be arrived at merely from a consideration of the mechanism of fracture as a result of prolonged tensile stress.

Since it is well known that at any given temperature the tendency for crystallization rapidly increases with increase in amount of deformation it is clear that with stresses resulting in minute amounts of strain the load must constantly be increased in order to maintain a constant strain rate (creep) as the metal exhibits increased resistance to elongation by virtue of the strain. The writer has previously drawn attention to the profound effects of elongation of the order of 0.02 per cent on rapidly decreasing further rates of creep.[3] Moreover, such minute permanent strains are not readily relieved by temperature softening (recrystallization or grain growth—boundary migration). When the amount of strain, however, is such as to promote temperature softening (at temperature of test) in order to maintain and not exceed this constant higher rate of creep it is necessary progressively to decrease the strain.

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[1] Associate Professor of Metallurgy, The Pennsylvania State College, State College, Pa.

[2] A. E. White, C. L. Clark and R. L. Wilson, "Influence of Time on Creep of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 167 (1935).

[3] C. R. Austin and J. R. Gier, "Comparative Studies on Creep of Metals Using a Modified Rohn Test," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. of Metals Division, Vol. 111, November, 1934, pp. 53–74.

It would be interesting in this connection if the authors could introduce into their analysis the difference in the *total amounts* of deformation obtaining along the various curves drawn for the three rates plotted—1.0 per cent, 0.1 per cent and 0.01 per cent per 1000 hr.

Again the authors are to be congratulated for the careful attention to the microstructural characteristics and changes observed in their series of tests. The progress of spheroidization is beautifully shown, but there appears to be considerable doubt on their interpretation of structures exhibiting recrystallisation. To the writer it appears that many of the structures for example (Fig. 8 (*c*)) exhibit evidence of lack of crystal change. Despite this absence of crystal deformation (other than elastic and submicroscopic plastic strain) it may be suggested that the elongation noted during test was wholly due to the breakdown of cohesion around the grains. This parting is then the factor which permits the oxidation to progress, as shown in the photomicrographs. In support of this hypothesis the writer suggests that unstressed steel held for the time at temperatures as indicated in the photomicrographs would not exhibit this intercrystalline oxidation, or at least not to anything like the same extent.

The writer regrets to note what he considers the confusion of terms in discussing recrystallization and equi-cohesion. Whatever may be one's conception regarding the nature of the phase which surrounds the grains in metallic crystalline aggregates there appears ample evidence to indicate that the grains themselves deform by progressive slip along certain preferred crystallographic cleavage planes. This is usually referred to as plastic deformation. Deformation in the grain boundaries, however, partakes of the nature of viscous flow where the time factor is paramount. In plastic strain-hardening within the grain the resistance to further deformation progressively increases unless the temperature is sufficiently high to promote grain growth or recrystallization, and it has been demonstrated that with minute amounts of strain grain growth occurs (boundary migration) rather than recrystallisation (formation of new grains from nuclei).

The mechanism of viscous flow is quite different. Strain apparently does not appear to inhibit further strain and the time factor is of the utmost importance.

Assuming that the concept of equi-cohesion has a real theoretical foundation, the term equi-cohesion temperature can only be associated with the rate at which the tension test is conducted. Thus much higher temperatures could be attained where transcrystalline fracture would occur with rapid elongation than would be the case if fracture resulted from many days or months at test.

The writer believes that it is evidence of this nature which the authors of the paper are accumulating and it would appear that if we are to obtain conclusive evidence to assist in interpreting what may be regarded as the peculiar behavior of the steels recorded, it is urgently necessary to begin an attack on the study of single crystalline material.

MR. T. S. FULLER.[4]—The observation by the authors that the fracture of the creep specimens was intercrystalline without evidences of strain-hardening in the vicinity of the fracture confirms those of other investigators in similar fields. Polycrystalline aluminum specimens tested and examined by Hansen and Wheeler[5] failed with intergranular fractures after being under load 220 hr. at a temperature of 250 C. Slip planes were not visible in the grains adjacent to the fracture and the ductility as measured by the reduction of area was low.

I have observed similar intergranular fractures in specimens of copper and iron which had been under sustained loads at elevated temperatures for hundreds of hours. Messrs. White, Clark and Wilson, therefore, have apparently touched upon a property common at least to many metals, namely, the faculty of failing under the proper conditions of prolonged stress and temperature with intergranular fractures exhibiting little reduction of area.

MR. C. L. CLARK.[6]—We appreciate the comments of Messrs. Austin and Fuller and are pleased to learn that in general they are in agreement with our findings.

Mr. Austin has raised several interesting points. First, referring to the irregularities in the curves of Fig. 2, the authors do not feel that too regular curves should be expected. It must be realized that two opposing tendencies, strain-hardening and recrystallization, are in progress, and the curve, representing the net effect of these two tendencies, must of necessity contain irregularities. If it be conceded that strain-hardening first occurs then the creep rate would tend to decrease. The recrystallization which then results increases the creep rate and thus the low and high points of the curve are produced.

Mr. Austin questions the conclusions regarding recrystallization of certain of the specimens. His question may be justified on the basis of the reproduced structures given in the paper but we are sure he would agree with us if the original microstructures had been available to him. Furthermore it is difficult to conceive how the resulting structure could be strain free, possess a lower hardness, and in certain cases possess a smaller grain size than the original structure if recrystallization had not occurred.

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[4] Metallurgist, Research Laboratory, General Electric Co., Schenectady, N. Y.

[5] D. Hansen and M. A. Wheeler, "The Deformation of Metals Under Prolonged Loading, Part I," *Journal*, Inst. Metals (British), Vol. 45, No. 1, pp. 229–264 (1931).

[6] Research Engineer, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

Mr. Austin likewise questions the use of the term "equi-cohesive temperature." He does, however, agree that at some particular temperature the mechanism of deformation changes from a transcrystalline to an intercrystalline phenomenon. We are, therefore, in agreement as to the influence of temperature and time on the mechanism of deformation and differ only as to the terms applied. We are convinced, however, that under sufficiently prolonged testing periods the equi-cohesive temperature is the dividing line between deformation occurring within the grains and that occurring around the grains and also that equi-cohesive temperature is the same as the lowest temperature of recrystallization. Considerably more evidence is being accumulated on this subject and will be presented in future papers. We have definitely found, however, that under prolonged testing times, the grains are always severely strained when the fracture is transcrystalline, and strain free when the fracture is intercrystalline.

We likewise wish to thank Mr. Fuller for the substantiating evidence presented for intergranular fractures in other types of metals and alloys.

A DIRECT-LOAD CREEP-TEST MACHINE

By J. J. Curran[1] and F. H. Morehead[2]

Due to the insistent and growing demands by industry for increased pressures and temperatures, and the difficulties and delays encountered in developing accurate data on the properties of materials of construction under these conditions of temperature and stress, it is not surprising that the engineer often is forced to design with factors of safety which are largely guess-work, and hence, often excessive.

Short-time tests at elevated temperatures take only a short time to complete, and the tests are relatively easy to perform, but the data thus obtained have a limited usefulness. Long-time stress-temperature-deformation or "creep" tests give much more definite and usable information, but they require a long time to complete. Extreme accuracy of temperature control and strain measurement are imperative if the results are to be of real value. Owing to the difficulties and time limitations involved, the field of high-temperature properties has as yet been only scantily explored.

Published reports indicate that much elevated-temperature data being currently developed are not reliable because of obvious faults in the methods of testing employed. Results shown by different laboratories for the same type of material often vary widely. Even though differences noted are due as much to non-uniformity of materials as to variations in testing technique, nevertheless, informed investigators do not hesitate to condemn as unreliable much of the data obtained currently, and methods which only a few years ago were considered entirely adequate.

A paper by Gillett and Cross[3] goes into creep testing methods in great detail, discussing the need for accuracy and how it may be achieved, and pointing out sources of possible error which are often neglected, or whose importance is not realized.

In recent years much discussion has been recorded on the subject of rapid methods for determining the rate of deformation in metals at constant temperature and under constant load. Differences of opinion exist as to the minimum testing period in which a reasonably accurate determination may be made, and as to the extent to which it is permissible to extrapolate the results obtained in this minimum period. Our experience leads us to believe that the closer we approach to constant loading and constant temperature, over a reasonably long period of time, the more reliable our creep data will be. A minimum period, we believe, would be 2000 hr., where truly sensitive and accurate temperature control and strain measurements are used, and longer periods where control and measurements are less accurate.

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[1] Research Metallurgist, Walworth Co., Greensburg, Pa.

[2] Vice President in Charge of Engineering, Walworth Co., New York City.

[3] H. W. Gillett and H. C. Cross, "Obtaining Reliable Values for Creep of Metals at High Temperatures," *Metals and Alloys*, July, 1933, p. 91.

This 2000-hr. period does not include the time for heating up, without load, to establish equilibrium in the specimen. It has been our experience that a steel specimen will not reach thermal equilibrium until heated for a period of approximately 100 hr. During this period the temperature distribution within the specimen changes almost constantly, and length changes also occur for about this period after the furnace and specimen have apparently reached temperature.

Changes apparently continue to take place within the bar until it acquires a stable coating of oxide, after which the heat-absorption properties, the temperature distribution and the length become constant for that temperature. Not until then can a specimen be loaded if the utmost accuracy is desired. Obviously, this phenomenon must be considered in connection with some of the "quick creep" tests.

We have endeavored to develop our elevated temperature testing practice to the highest attainable accuracy, and at the same time make it as convenient and fool-proof as possible. We have drawn freely from the experience of others, making modifications to include features which seemed desirable. The A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22–35 T)[4] has been followed closely.

Room Temperature Control:

In view of the basic design of our elevated-temperature equipment, it was felt unnecessary to house it in a truly constant-temperature room. For convenience it was placed in the main laboratory, which is heated by electrically driven unit heaters, and the temperature controlled within narrow limits by a thermostat placed near to and at the same elevation as the creep furnaces.

The room is thus kept at a temperature of about 85 F. during the major portion of the year. During a few months of summer, when outside temperatures are high, this temperature is exceeded at times, but it is thought that the methods are not sufficiently susceptible to error from this temperature variation to warrant automatic cooling.

Furnaces for Creep Tests:

Test specimens are heated chiefly in conventional resistance furnaces of the gap-wound type or the tapped-and-shunted type.

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1291 (1935); also 1935 Book of A.S.T.M. Tentativ Standards, p. 1415.

These furnaces heat by radiation and conduction. Owing to the fact that the grips or shackles are large, as compared to the specimen itself, and since they extend out of the furnace at each end and carry away heat by conduction, it is necessary to concentrate the heating effect in the ends of the furnace, in the shackles themselves more than in the test specimen. By thus compensating for the heat losses and end effect, and by distributing the temperature in the central portion by means of a metallic sleeve of silver inside the refractory chamber, it is possible to obtain a high degree of uniformity in the test specimen.

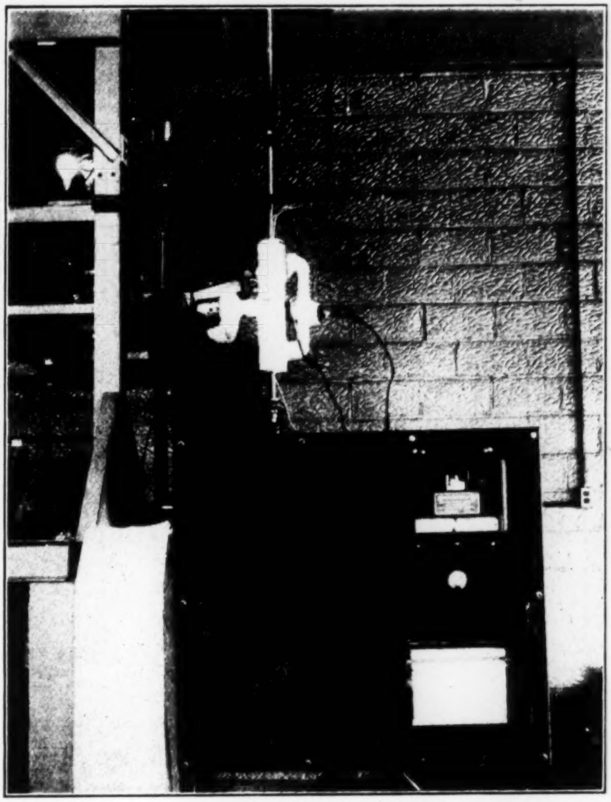
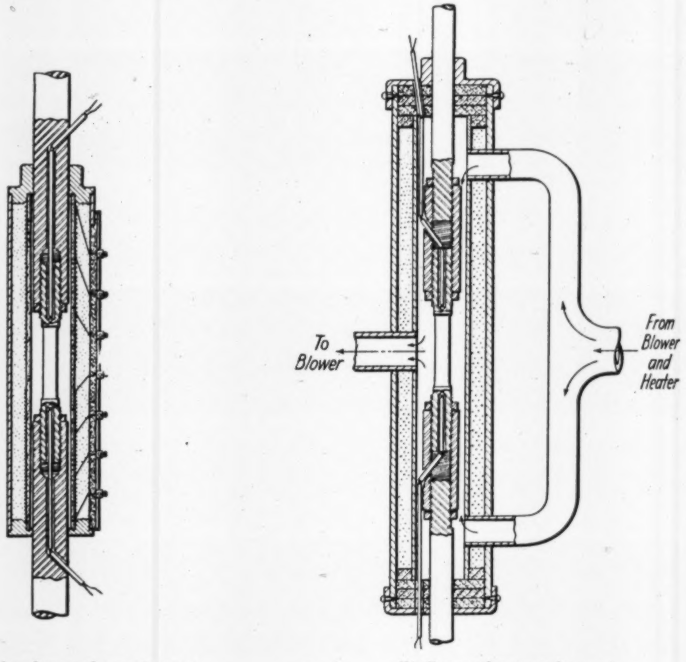


FIG. 1.—Forced Convection Creep Test Furnace.

Extreme precautions must be observed in the use of these furnaces, but with proper care they will provide the desired accuracy and uniformity of temperature throughout the gage length. After calibrating a furnace for a given temperature, it is essential that the test specimen proper be located at exactly the same point as the calibrating specimen previously occupied. For best results it is necessary, also, that the metallic sleeve inside the furnace be kept properly shaped to fit the refractory closely.

There are indications that slight errors may also exist due to lack of absolute interchangeability between the calibration specimen and the test specimen proper. It is obvious that the temperature gradients within and along a drilled calibration specimen cannot be exactly the same as in a solid specimen.

These furnaces attempt to heat uniformly by direct radiation and conduction a system comprised of non-uniform sections, with portions of that system extending outside the furnace. By means of the methods of compensation employed, reasonable uniformity of temperature distribution is achieved. Slight changes in the position of the specimen, or the testing of specimens of widely different thermal conductivity, however, introduce errors which are difficult to detect and compensate. Other errors may be produced by the fact that several auxiliary couples extend from the furnace during calibration, but are not present when the actual test specimen is in place.



(*a*) Conventional tapped type furnace. (*b*) Convection type furnace

FIG. 2.—Creep Test Furnaces.

We feel, in this connection, that the A.S.T.M. Tentative Method of Test E 22 – 35 T recommendation that the inside diameter of the furnace be made as small as possible, merely large enough to allow for shackles and extensometer, might well be revised. We are convinced that a larger inside diameter would promote greater uniformity of temperature distribution.

We have spent some time on the development of a furnace designed to eliminate some of these sources of error (see Fig. 1). In this furnace the principle of forced convection is used. The atmosphere of the furnace is continuously recirculated around the specimen and shackles, heat being applied at a remote point by passing the circulating air through a heating chamber. This arrangement involves some mechanical difficulties, but promises to give the maximum of temperature uniformity, with great ease of control. Due to the application of the convection principle, it is felt that errors due to the shape, location, and thermal conductivity of the test specimen will be reduced to a minimum. The principle involved deserves careful study by all interested in creep and short-time tests.

In Fig. 2 are shown diagrammatically, for comparison, a conventional tapped type furnace and the convection type which we are investigating.

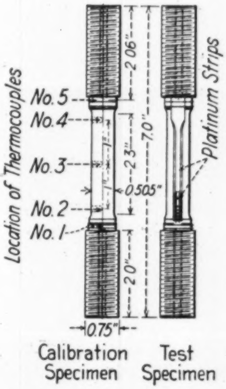


FIG. 3.—Test Specimens.

It should be noted that in the latter furnace we have cut down the size of the shackle rods to minimize conduction of heat away from the specimen and out of the furnace.

While not indicated in the diagram, it is necessary to lag the furnace and the air conduits heavily, though this may be minimized by utilizing a shell and conduits of insulating materials. There is some question as to whether the furnace lining should best be metallic or of insulating refractory material. Further work will doubtless answer this question.

The effect of atmospheres on the test specimen is difficult to determine in the ordinary type furnace, but in the convection furnace it is a relatively simple matter to control the atmosphere or to investigate special atmospheres.

To illustrate the uniformity of heating in different furnaces, the calibration results obtained on cast monel metal in two gap-wound furnaces and in a convection furnace are given in the following tabulation. The two gap-wound furnaces were made up identically. Furnace A, however, gives a very poor performance, while its twin, furnace C, gives excellent results, comparable with those in the convection-type furnace E.

THERMOCOUPLE LOCATION[a]	FURNACE A GAP WOUND	FURNACE C GAP WOUND	FURNACE E CONVECTION
No. 1	702 F.	702 F.	703 F.
No. 2	703 F.	702 F.	701 F.
No. 3	710 F.	703 F.	700 F.
No. 4	716 F.	705 F.	700 F.
No. 5	723 F.	703 F.	702 F.

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[a] See Fig. 3.

In these tests furnaces A and C were controlled from the couple at point 5, and furnace E was controlled from a couple situated outside the specimen, opposite the midpoint of the gage length and about $\frac{3}{8}$ in. from the specimen.

Test Specimens:

The test and calibration specimens used, as shown in Fig. 3, conform to those described by Gillett and Cross.[3] Their platinum-strip method of measuring elongation is also utilized. The telescoping platinum strips are marked with crosses or X's, by means of a razor blade or a carefully selected needle. When carefully made, the X's furnish reference points to which the cross hairs of the filar microscope may be set with accuracy.

Temperature Control:

Control of temperature is achieved by means of a chromel-alumel thermocouple inserted into a drilled hole extending axially through the threaded end to the shoulder, just outside the fillet. This couple, connected to a suitable potentiometer controller, with provision for anticipatory action, increases and decreases the current in the furnace winding, and maintains the temperature of the specimen constant within very narrow limits. Another couple, located similarly in the other end of the specimen, is connected to a potentiometer recorder and furnishes a continuous record of temperature fluctuations within the specimen. Periodic checks are made on the specimen by means of a precision potentiometer, by disconnecting momentarily the controller and the recorder. These periodic temperature readings are taken as the actual temperature record of the test. A separate controller, of course, is furnished for each furnace.

Controlling from the axis of the specimen is obviously not the best practice, but it is fairly satisfactory when suitable means of anticipatory control are utilized. In our convection furnace, control is external, and no anticipation is required.

In connection with the periodic temperature checks, it was early observed that in making and breaking connections, as by plugging in, some temperature fluctuations resulted. Consequently, it was found necessary to discard the standard "compensating" leads usually furnished for chromel-alumel systems and to replace them with lead wires of chromel-alumel. All other metals were eliminated from the circuit between the furnace and its control and recorder by making up suitable switching plugs of these metals. This resulted in the elimination of appreciable errors from unavoidable localized changes in temperature.

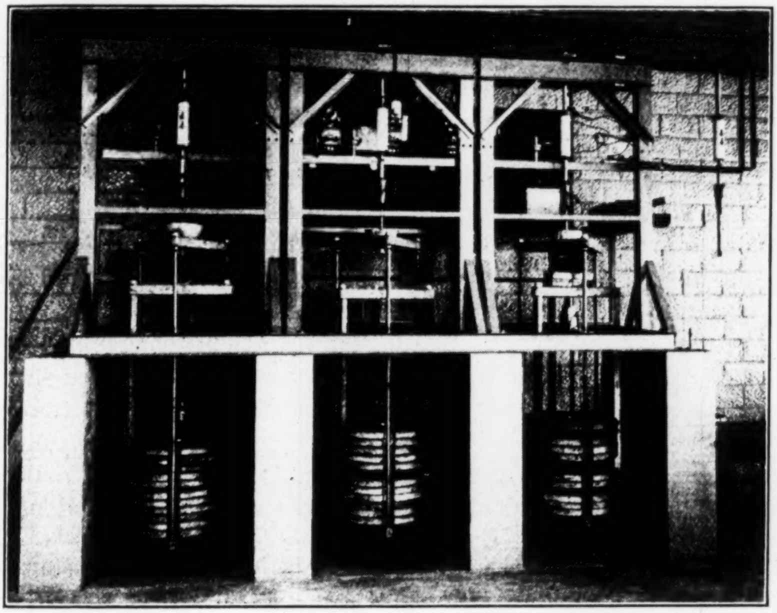

FIG. 4.—General View of the Machine Showing Method of Loading.

To minimize errors caused by variations in electromotive force with changes in room temperature, the dry cells used in connection with the potentiometers are kept at a constant temperature, slightly above that of the room, in a thermostatically controlled chamber. This precaution minimizes manual rebalancing of potentiometer and cold end compensator circuits.

Loading:

A general view of the machine showing the method of loading is shown in Fig. 4. A welded framework of structural steel rests on concrete piers which are independent of one another. Separate steel frames carry each furnace. The test specimens and furnaces are supported directly on the framework with one ball-and-socket support at the top and another in the turnbuckle below the specimen. A triangular member is attached to the turnbuckle and carries a three-rod suspension holding the weights which load the specimen. A similar suspension holds the extra weights, when not in use, by means of pins inserted through its vertical members. The idle and loading suspensions are not in contact in any way. Weights are of galvanized cast iron and weigh about 250 lb. each. They are accurately weighed on calibrated scales and each is stamped with its true weight. Space has been provided for a sufficient number of weights to apply a 30,000-lb. unit load on a standard specimen. A small hydraulic jack with micrometer lowering device is used to apply and remove the load (note jack in place on right-hand unit).

Space has also been provided for testing a specimen with a 24-in. gage length when and if a means can be provided for maintaining a uniform temperature in a specimen of this length. The convection furnace appears to offer a means of attaining uniformity in such unconventional specimens.

A calibration station at the extreme right (Fig. 4) is used for calibrating new furnaces, thus avoiding loss of time at testing stations.

While this type of creep-test machine has one marked disadvantage in that it occupies considerable floor space, it has certain advantages over the lever type. A lever system oscillates with each slight change in the temperature of the furnace, due to change in length of specimen and shackles. No lever, knife edge, or seat is perfect. Even though it were perfect at no load, it would not be so under load. Knife edges (six of them) cannot all be made the same shape, and practical knife edges are not frictionless. Errors in alignment are present in every lever and mounting, and localized temperature changes affect them.

In the lever-type machine, about 30 in. of the frame are included in the stressed portion of the mechanism. Unless great care is used to control room temperature, changes in length of this element must be reflected in the specimens under test. Localized heating by the furnaces themselves is difficult to avoid.

The direct-load machine requires no leveling of the beam, nor can it introduce errors due to eccentric loading. It is essentially frictionless.

Measurement of Deformation or Strain:

Measurements of elongation of the creep specimen are made by means of a micrometer microscope, sighted through a quartz window on reference marks on the platinum strips attached to the shoulders of the specimen. The microscope is graduated to read easily to 0.00005 in., affording a sensitivity of 0.0022 per cent or 0.000022 in. per inch on the 2.3-in. gage length used. The platinum strip method of measuring deformation of the specimen introduces an unimportant error due to the difference in the coefficient of expansion of the platinum and the material under test. The platinum strip is attached to the specimen only at the ends and hangs free of the specimen and parallel to the gage length. In this position it can introduce a further error because of its small mass, which permits it to heat up and cool down faster than the specimen itself. This error is probably not great enough to cause serious difficulty, but is eliminated entirely in the convection type of furnace with which we are working.

The use of a single microscope for the measurements does eliminate the effect of variations in room temperature outside the furnace, and in addition is more convenient than some of the twin microscope assemblies.

The micrometer microscope is calibrated against a certified micrometer slide, and is checked periodically against a stage micrometer.

Acknowledgment.—Grateful acknowledgment is made of the advice and cooperation extended by the personnel of Battelle Memorial Institute.

SOME CREEP TESTS ON LEAD AND LEAD ALLOYS

By Albert J. Phillips[1]

Synopsis

Creep test data on lead and lead alloys from tests of three years' duration are presented to show that it is unsafe to extrapolate creep rates calculated from short-time tests. Many alloying elements which increase the tensile strength of lead decrease the resistance to creep under low loads. Copper added to lead improves the creep resistance very markedly. Antimony lowers the creep resistance except in the age-hardened condition where the elongation under fairly high tensile loads is practically nil. However, such metal will fracture without flow under sustained loads, making it difficult, if not impossible, to calculate a safe working stress. Tin likewise lowers the creep resistance of lead but in the heat-treated condition the alloy shows excellent creep resistance without serious loss in ductility. Tellurium added to lead lowers its creep resistance under low loads in both the hard-rolled and annealed condition.

There is a growing appreciation by engineers, particularly as metals are used at higher temperatures, that tensile strength data, as normally obtained, are not necessarily reliable indications of the service characteristics of metals. Tensile strength and even conventional yield point values many times the serviceable strength are obtained if tests are conducted at or above the recrystallization temperature. For this reason testing engineers are now undertaking the laborious task of determining "long-time tensile strengths" or "creep strengths" of various alloys over their useful temperature range.

The magnitude of this task is hard to comprehend and the difficulties, particularly where the tests are at high temperatures, are disheartening. For example, a testing engineer is requested to predict accurately what load a certain alloy will support over a period of 10 yr. without yielding say more than 1 per cent when operating in a given temperature range. Obviously the testing engineer cannot conduct 10-yr. tests to answer the question. Yet the question must be answered fairly accurately, for the designing engineer of today is not content to use the generous "factors of safety" prevalent a few years ago.

The testing engineer has solved this dilemma by a compromise involving a fundamental assumption. Creep tests are now conducted for short periods of say 2000 hr. but measuring the strain with great accuracy, the creep rate computed, and on the assumption that the creep rate of a given metal is a constant at a given temperature, the rate is extrapolated to the time desired. The assumption seems logical particularly where it can be shown that the strain-time curve has become a straight line, but, like all extrapolations, it may be an extremely inaccurate proceeding and one that needs repeated checking before it can be used with confidence.

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[1] Superintendent of Research, Research Dept., The American Smelting and Refining Co., Maurer, N. J.

Such checking is not simple particularly with those tests involving temperatures of over 1000 F. since they involve tests lasting years and conducted on a multiplicity of specimens. In spite of these difficulties the Joint Research Committee of the A.S.M.E. and A.S.T.M. on effect of Temperature on the Properties of Metals, has initiated 25,000-hr. tests on certain steels. Results from these long-time tests will not be available until 1938.

Fortunately, certain common metals, for example, lead, tin and cadmium, behave at room temperature in a manner analogous to the stiffer structural material at elevated temperatures. A study of long-time creep results obtained with these metals may serve a double duty in fomulating generalizations applicable to other metals more difficult to test. Four years ago creep tests on the low-melting metals and some of their alloys, particularly of lead, were started in the Central Research Laboratory of the American Smelting and Refining Co. Some of the original specimens are still under test and will be continued until they have extended at least 2 per cent.

SPECIMENS

It was decided to conduct all tests on wrought materials for several reasons: first, the metals in question were used most commonly in the wrought form; second, the coarse structure of many cast lead alloys would necessitate the use of castings of large cross-section if the orientation effects of individual crystals were to be minimized; third, fabrication not only breaks up the coarse cast crystalline structure but also closes up blowholes, shrinkage voids, and even reduces the detrimental effect of dirt inclusions; and, fourth, incidental heat treatment in the cast structure is eliminated or controlled in the homogenized wrought metal.

All of the specimens reported herein were prepared in the following standard fashion: The metals were melted in either clay-graphite crucibles or cast-iron pots and suitably super-heated to a temperature of 400 C. and poured rapidly into an open cast-iron mold preheated to 100 C. The bar obtained thus was 10 in. long, $1\frac{5}{8}$ in. wide and $\frac{3}{4}$ in. thick. Minor defects on the surface of these bars were carefully removed by hand scraping and they were then rolled to size. In rolling, the bars were not rolled rapidly enough to cause them to heat up appreciably. The castings were not cross-rolled and initially the passes were each 0.04 in., but when the specimen had been rolled to 0.40 in. the reduction per pass was thereafter maintained at 10 per cent. In most cases the specimens used were 0.10 in. thick but the width was varied from 1.5 to 0.5 in. Tests proved that reduced sections were unnecessary to obtain breaks consistently within the gage lengths and consequently the rolled strips were prepared very simply by clamping them between wood blocks and planing the edges with a sharp wood plane. By this procedure remarkably straightsided specimens with minimum distortion by cold working could be prepared. The over-all length of the specimens was 14 in. with a gage length of 8 in.

Practically all the metals and alloys tested were prepared in the "as-rolled" condition and in the "annealed" condition. The "annealing" consisted of heating the fully prepared specimen to 100 C. for 1 hr. Many of the alloy specimens and a few pure metals were also tested in the "quenched and age-hardened" condition which consisted of heating to 225 to 230 C. for 2 hr., quenching in an ice-brine solution and aging at room temperature for 1 yr.

APPARATUS

The apparatus used was fundamentally very simple and sufficiently inexpensive to permit the installation of a large number of stations. The specimens were hung vertically on $6\frac{3}{4}$-in. centers from a $2\frac{1}{2}$-in. horizontal angle iron, their upper end being clamped between the side of the angle iron and a piece of flat steel $2\frac{1}{2}$ in. long, $1\frac{1}{2}$ in. wide and $\frac{1}{4}$ in. thick by means of a 2-in. malleable iron C clamp. The load on the specimens consisted of a lead weight suspended by a short length of chain from a central pin between the lower grips. The latter consisted of two flat pieces of iron 3 by $1\frac{1}{2}$ by $\frac{1}{4}$ in. with a No. 12 hole drilled in the center $\frac{1}{4}$ in. from one end. A 2-in. C clamp held the grips in place.

The extension of the specimen was measured with a pair of dividers. This gave an accuracy of 0.01 in. in 8 in. or 0.00125 in. per inch. Since it was contemplated that the tests would be continued for many months, need for greater accuracy was not anticipated. Readings on specimens heavily loaded were taken hourly while lightly loaded specimens were read as infrequently as once a month.

The tests were conducted at room temperature, which averaged 24 C. with extremes of 17 and 28 C. The room was reasonably free from excessive humidity and corrosive fumes so that it was felt that corrosion of the specimens did not influence the tests. In no case was there any indication of corrosion deposits or formation on the samples. The test location was remarkably free from vibration, for even the daily passage of a switching engine a short distance from the building did not cause detectable vibration of the specimens.

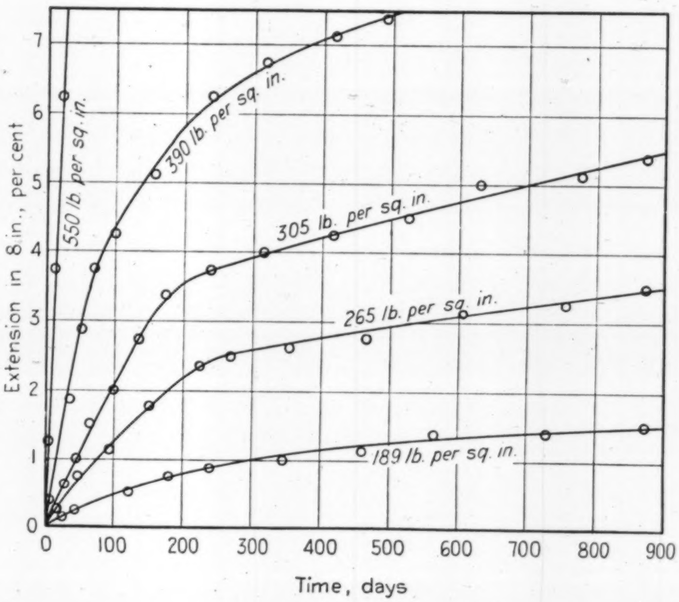

FIG. 1.—Creep Test Data on a Commercial Lead at Room Temperature.

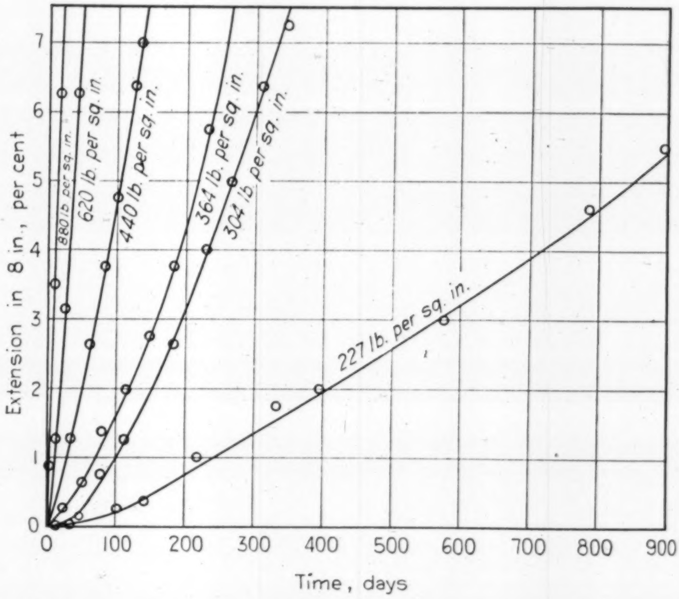

FIG. 2.—Creep Test Data on a Commercial Lead with 1 per cent Antimony at Room Temperature.

DATA

While the collection of creep test data was extremely easy and simple, the presentation of such data in a form that will be useful is beset with complications. The data were first plotted in extension-time curves for each individual test specimen, the individual curves being labeled with their tensile load in pounds per square inch. Since from four to six specimens with different load values were used for each metal being tested, it was found advisable to plot only one alloy per chart to avoid confusion. Figure 1 illustrates the method of plotting fundamental data on a commercial lead having the following analysis: Silver, 0.0005 per cent; copper, 0.0004 per cent; iron, 0.0002 per cent; zinc, 0.0005 per cent; cadmium, 0.0006 per cent; antimony, 0.0005 per cent; bismuth, 0.042 per cent; and lead (by difference) 99.95 per cent.

This type of graph is fundamental and is a necessary preliminary in comparing creep data on various alloys. However, an inspection will demonstrate that it is difficult to compare and evaluate different alloys from charts of this sort, particularly when data from different sources and different unit load values are being considered. Consequently, from the data plotted in extension-time curves, an attempt was made to determine creep rates which could be plotted against tensile loads yielding a single curve for each alloy. Where the extension-time curves were practically straight lines, this procedure was both simple and accurate. Fortunately, most tests on commercial leads resulted in reasonably straight extension-time curves. Yet Fig. 1 illustrates a lead which showed a marked decrease in rate of extension with time. A great many lead alloys have curves of this type. Other lead alloys, particularly those capable of retaining work-hardening, have curves of the opposite type as shown in Fig. 2 in which the rate of extension is initially low but increases as the test progresses. The composition of this alloy was as follows: Antimony, 0.96 per cent; copper, 0.065 per cent; silver, 0.0005 per cent; iron, 0.0002 per cent; zinc, 0.0004 per cent; cadmium, 0.0001 per cent; bismuth, 0.11 per cent; and lead (by difference) 98.86 per cent.

It is evident that a creep rate curve representing either Figs. 1 or 2 will be, at best, a compromise. However, considerable experimenting with the data has shown that if, instead of attempting to plot a single point to represent the rate of extension, a horizontal line is plotted, the extremes of which represent the maximum and minimum rates of extension that have been experienced over a definite limited period and a point on the line indicating the average rate of extension, a reasonably satisfactory curve may be drawn through the values thus plotted. All the rate curves shown in this paper were derived by this procedure as illustrated by Fig. 3.

It has been found most convenient to plot the reciprocal of the rate of extension on a log scale against the log of the tensile stress. This method of plotting has the advantage that extrapolations can be clearly indicated and their significance readily recognized. In plotting the data of this paper the maximum, minimum and average rate of extension for extensions of 5 per cent or for periods of not less than 1000 days were used. In those cases where the actual extension of the specimen was less than 1 per cent up to date, the fact that the rate was extrapolated is clearly indicated by the use of a broken line.

For convenience in comparing various metals and alloys, the tensile strength was plotted as the terminal point along the ordinate axis for each curve in spite of the fact that this axis represents a rate of extension of 1 per cent per 0.01 day whereas the rate of extension during the tension tests was actually 0.05 in. per in. per min. In Fig. 3 the curves represent data from two sets of specimens which were identical in every respect except one set of specimens was tested "as rolled" and the other "as annealed". The analysis of the metal was as follows: Silver, 0.0007 per cent; copper, 0.0009 per cent; iron, 0.0003 per cent; zinc, 0.0006 per cent; antimony, 0.0003 per cent; bismuth, 0.030 per cent; and lead (by difference) 99.96 per cent. It is evident that a fairly satisfactory rate curve can be obtained even from data showing considerable differences in rate of extension.

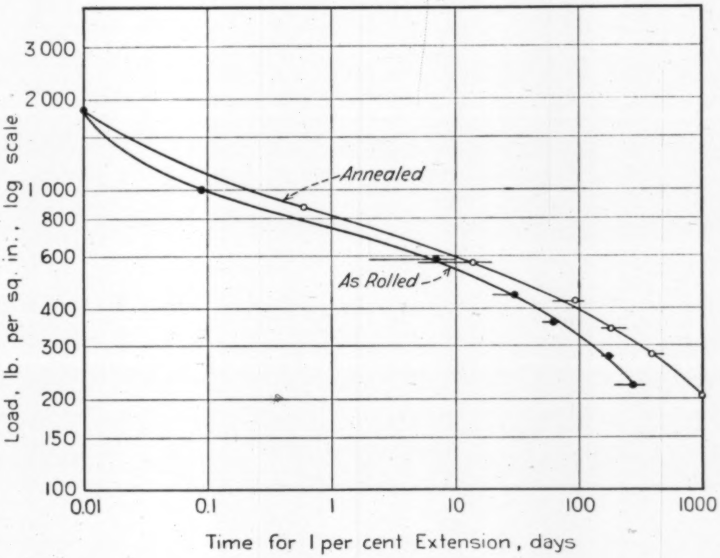

FIG. 3.—Creep of a Commercial Lead at Room Temperature.

Tests have been conducted on 10 different commercial brands of desil-

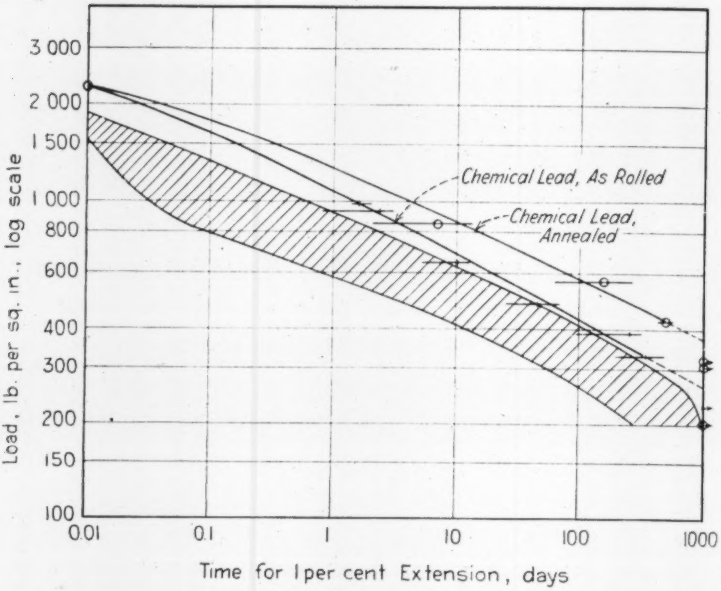

FIG. 4.—Creep of Common Lead and Chemical Lead.

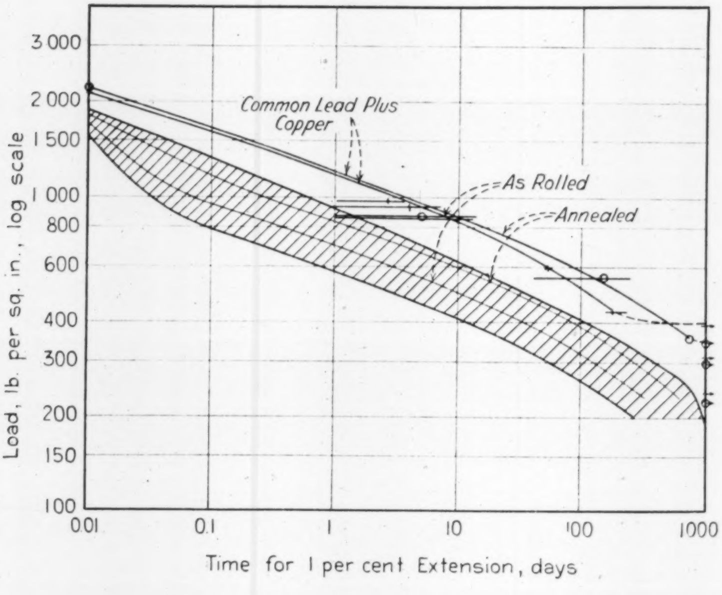

FIG. 5.—Creep of Common Lead and Common Lead Plus 0.065 per cent Copper.

verized lead using 22 different sets of specimens. While considerable variation was experienced from one test to another, it was impossible to associate these variations with either the composition or purity of the lead or even with the preparation, although the data from "annealed" samples was more consistent than those obtained from the "as rolled" specimens. Upon plotting all of the rate curves for desilverized lead upon a single chart it was found that the creep rates all lie within the shaded band shown in Fig. 4 and all subsequent figures. Tests upon chemical lead (A.S.T.M. Grade II)[2] gave results which do not lie within the shaded band in Fig. 4 and are represented by the two curves above this area. The analysis of the particular chemical lead tested was as follows: Silver, 0.0057 per cent; copper, 0.0553 per cent; iron, 0.0004 per cent; cadmium, 0.0006 per cent; antimony, 0.0004 per cent; nickel and cobalt 0.0043 per cent; bismuth, 0.0005 per cent; and lead (by difference) 99.93 per cent.

The greater creep resistance of chemical lead over refined desilverized leads is unquestionably due to the stiffening effect of copper always found in chemical lead. Thus in Fig. 5 the effect of adding copper to an ordinary common lead is illustrated. The two curves within the shaded band represent specific tests in the "as-rolled" and "annealed" condition on a common lead having the following analysis: Silver, 0.0005 per cent; copper, 0.0005 per cent; iron, 0.0002 per cent; zinc, 0.0004 per cent; cadmium, 0.0001 per cent; antimony, 0.0004 per cent; bismuth, 0.11 per cent; and lead (by difference) 99.89 per cent. The two curves above the shaded band in this figure represent data obtained with this same lead alloyed with 0.065 per cent copper. It should be noted that the arrows along the right-hand margin indicate specimens that are still under test and which have extended less than 1 per cent in a period of approximately 1200 days.

It is at once evident from Figs. 4 and 5 that leads containing copper have improved creep-resistance over copper-free leads. In fact, common lead alloyed with 0.065 per cent copper has the best creep resistance in the "as-rolled" condition of all the leads and alloys tested to date. This is not brought out as clearly in Fig. 5 as it is by the data from which the figure was constructed. For example one of the test specimens of common lead alloyed with copper has supported a load of 386 lb. per sq. in. for 1200 days without any detectable extension. As copper stiffens lead and raises its tensile strength, it might be considered only logical to predict that such stiffening would be accompanied by increased creep resistance. However, contrary to the general assumption, most elements that stiffen lead when alloyed with it impair rather than improve its creep resistance. Thus in Fig. 6 the two curves which merge together as they pass through the shaded band, represent creep test data obtained from samples in the "as-rolled" and "an-

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[2] Standard Specifications for Pig Lead (B 29 – 35), 1935 Supplement to Book of A.S.T.M. Standards, p. 26.

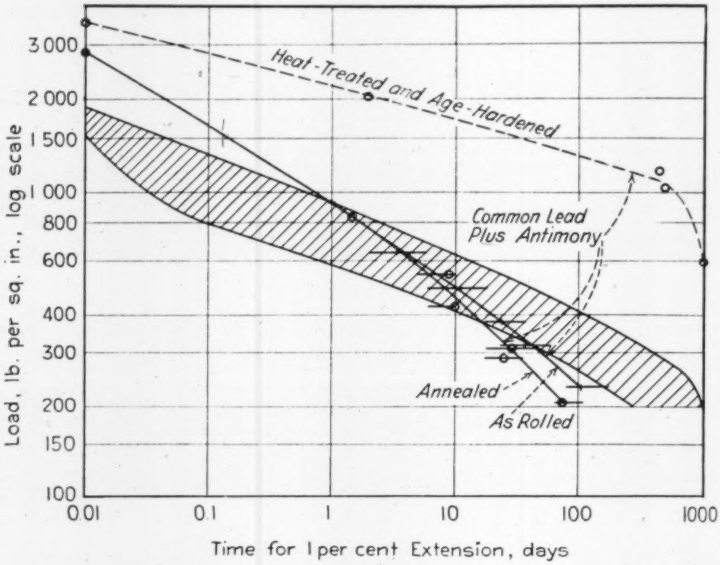

FIG. 6.—Creep of Common Lead and Common Lead Plus 1 per cent Antimony.

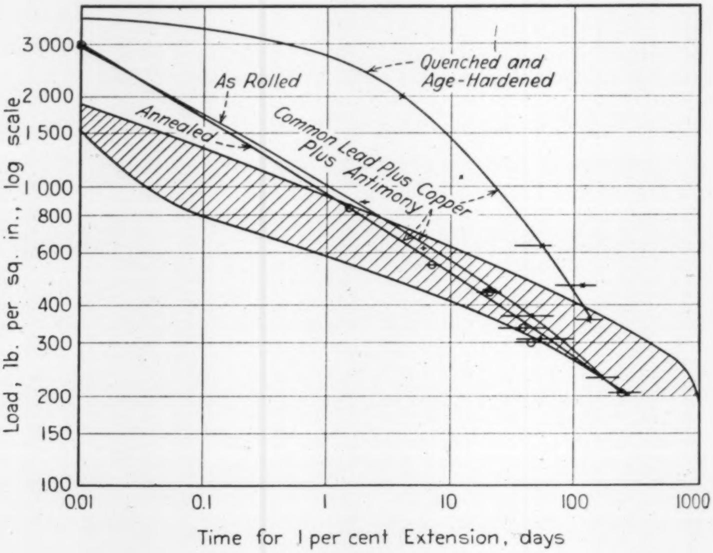

FIG. 7.—Creep of Common Lead and Common Lead Plus 0.065 per cent Copper Plus 1 per cent Antimony.

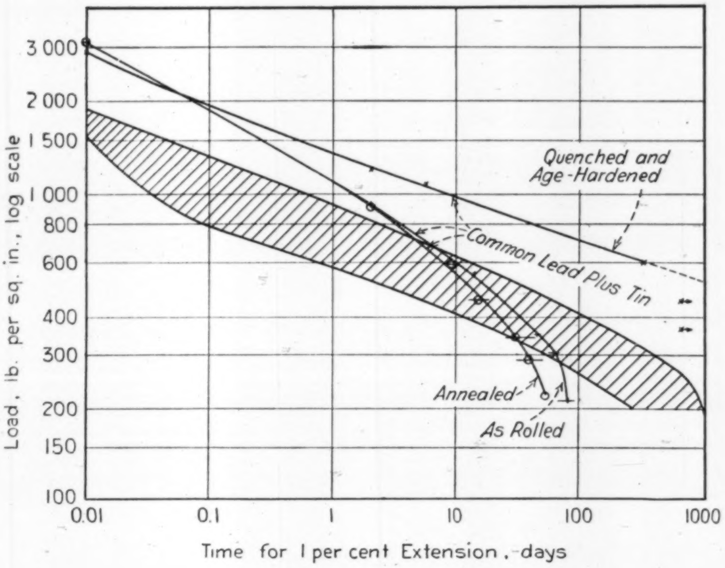

FIG. 8.—Creep of Common Lead and Common Lead Plus 3 per cent Tin.

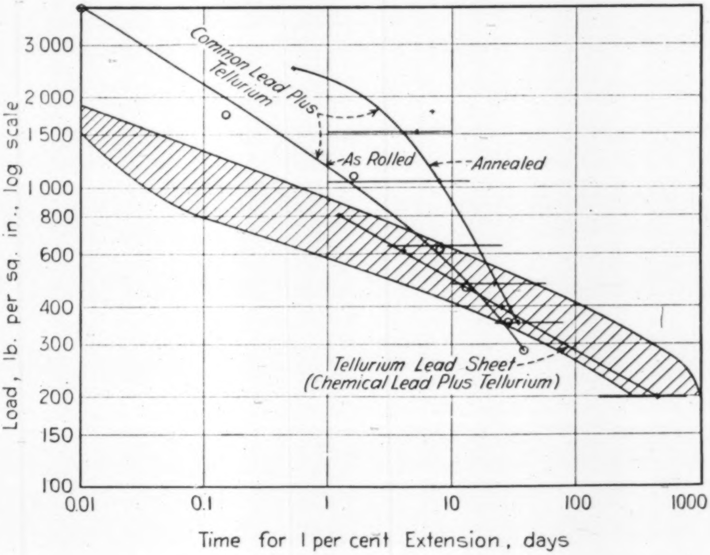

FIG. 9.—Creep of Common Lead and Common Lead Plus 0.055 per cent Tellurium.

nealed" condition prepared by alloying 0.95 per cent antimony with the same common lead represented in Fig. 5. The tests show conclusively that while the alloy is much stronger under heavy loads than common lead, it creeps with low loads decidedly more rapidly. The broken line in Fig. 6 represents the results of tests on the same alloy after heat treatment and "age-hardening." The line is dotted to indicate extrapolation for in no case did the specimens actually extend 1 per cent; three of the specimens have broken with less than 1 per cent elongation and the three remaining specimens have been under test for 2 yr. with less than 1 per cent extension. It should be noted that this alloy in the "as-rolled" and "annealed" conditions gives remarkable elongations in creep, usually over 100 per cent.

The addition of copper to an antimonial lead improves its creep resistance in the "annealed" and "as-rolled" conditions just as it does a common lead. Figure 7 illustrates creep tests on an alloy made from the same common lead as was used for the previously discussed alloys but with 0.065 per cent copper as well as 0.96 per cent antimony. It is evident that the addition of copper has improved the creep resistance under low loads, bringing it into the lowest range of creep resistance experienced for common lead. Copper has also changed the alloy in the "heat-treated and age-hardened" condition, increasing the ductility markedly but unfortunately lowering the creep resistance so that the values obtained with the lower loads are no better than those experienced for many commercial leads. Results obtained from a 1 per cent antimonial alloy compounded directly from chemical lead, which contained 0.055 per cent copper, were practically identical with those illustrated in Fig. 7.

Figure 8 illustrates the creep data obtained from tests on an alloy made from common lead and containing 3.23 per cent tin. The addition of tin to lead decreases the creep resistance under low loads in the "as-rolled" and "annealed" condition but increases the creep resistance in the "heat-treated and aged" condition but without the embrittlement experienced with antimonial leads. The broken portion of the heat-treated curve merely indicates that the specimens with loads below 600 lb. per sq. in. have not extended 1 per cent in the 2 yr. that they have been under test. This creep resistance of the heat-treated tin alloy is even more interesting when it is noted that the treatment has had little or no effect on the tensile properties. While the lead-tin constitution diagram would indicate that the alloy should be susceptible to such treatment, it is believed that this is the first demonstration that the lead-tin alloys are improved by heat treatment to produce precipitation hardening.

Tellurium exerts a remarkable effect upon lead according to Singleton and Jones,[3] increasing the temperature of recrystallization so that the alloy can be permanently work-hardened and thus imparting many desirable properties. Strangely enough, the increase in recrystallization temperature is not reflected in improved creep properties as might be assumed. Figure 9 illustrates the creep data obtained with two samples of tellurium lead. The two curves which start above and cross the shaded band were made by adding 0.055 per cent tellurium to common lead and were prepared in the "as-rolled" and "annealed" condition using standard technique. The curve lying entirely within the shaded band represents data obtained on specimens cut from a sample of commercial tellurium lead sheet. Unfortunately there was not sufficient sample to permit a complete chemical analysis but a spectrographic check showed that the alloy was made by adding tellurium to chemical lead. It is natural to assume that the improved slope of the curve for this alloy is largely due to the copper of the chemical lead.

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[3] W. Singleton and Brinley Jones, "Some Effects of the Addition of Tellurium to Lead," *Journal*, Inst. Metals (British), Vol. 51, p. 71 (1933).

DISCUSSION

This research was designed to facilitate the study of the physical metallurgical factors affecting creep rather than to prepare physical data to be used by designing engineers; consequently the excellent system of presentation of creep data advocated by McVetty[4] was not used. By the use of the creep rate curves illustrated in Fig. 3 it is possible to compare quickly a relatively large amount of test data and to permit deductions readily as to the relative merits of the alloys under test.

Comparing the data of this paper with those published by Moore and his associates,[5] there are certain outstanding discrepancies. Moore's values for commercial pure lead lie very nicely within the shaded band used to locate the range of values experienced for commercial leads in Figs. 4 to 9. However, for both the tin and antimony alloys his figures are intermediate between those shown in this paper for the specimens in the "as-rolled" and "annealed" condition and those obtained on the "heat-treated" specimens. This is not surprising since the specimens which Moore tested were cut from cable sheathing and could very well have been in an intermediate condition.

From the data accumulated to date it has been impossible to make any generalization with regard to the effect of various alloying additions, that will not have many exceptions. However, there is considerable evidence to indicate that alloying elements that form true solid solutions decrease the creep resistance of lead and probably in proportion to their solid solubility. Alloy structures which tend to interfere with slip either by general obstruction of a second phase at the grain boundaries or by precipitation of a second phase within the grains, decreases the tendency for lead to creep.

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[4] P. G. McVetty, "The Interpretation of Creep Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II. p. 105 (1934).

[5] H. F. Moore, B. B. Betty and C. W. Dollins, "The Creep and Fracture of Lead and Lead Alloys," University of Illinois *Bulletin No. 23*, Vol. 32 (1935).

CONCLUSIONS

1. It is certainly impossible to predict the creep rates of many lead alloys from tests of less than 1 yr. duration.

2. A considerable range of creep rates for commercial leads may be experienced, probably depending upon uncontrolled variations in the preparation of test specimens.

3. The creep rates of many alloys of lead vary tremendously with the fabrication and heat treatment.

4. Tension tests do not offer any indication of the creep resistance of lead alloys.

5. Improved creep resistance may be imparted by "precipitation-hardening" treatment even when such treatment has no apparent effect upon other properties.

6. By the addition of 0.065 per cent copper to refined desilverized lead, an alloy is produced superior in creep resistance in the rolled condition to most commercial leads and alloys.

Acknowledgment:

The work presented in this paper represents a part of a general program investigating the fundamental properties of lead and its alloys, instigated by Mr. Jesse O. Betterton, General Manager of Lead Refineries and Director of Research, The American Smelting and Refining Co.

DISCUSSION

Mr. B. B. Betty[1] (*presented in written form*).—The author has issued a word of warning about the use of short-time tensile tests in predicting service characteristics of metals. He might well have let this apply to the use of creep test data as well, for a good deal of judgment and clear thinking are necessary in the successful application of long-time test data to metals operating under service conditions. In the first place, creep tests may be made for two somewhat different purposes: first, to reveal the relative order of merit of a number of metals in resisting deformation under a long-continued steady load, and, second, to enable the designer to restrict creep within a predetermined maximum allowable limit.

Attention is called to the fact that the relative merit of two grades or alloys of lead with respect to creep resistance depends upon the stress to which they are subjected. Referring to Fig. 6 of the paper, one finds that at a stress of 2000 lb. per sq. in. a 1 per cent antimony-lead alloy required several times as long to creep 1 per cent as did common lead, whereas at 200 lb. per sq. in. the ratio of the rates of creep is reversed. One can say that metal B is better than metal C only when the conditions under which the metals are to be tested or used are also specified. This emphasizes the importance of selecting the conditions of a laboratory test with special regard to the service in which metals are to be used.

At the University of Illinois there is in progress an extensive investigation of the creep resistance of lead and lead alloys used for cable sheathing. The tests are made on specimens cut from extruded pipes and sheaths. In view of the fact that Mr. Phillips has obtained quite appreciable variations in creep resistances of rolled lead, due probably to slight differences in thermal and mechanical treatments, one cannot expect perfect agreement between his test results and those made at Illinois on metals subjected to entirely different thermal and mechanical treatments.

The writer's experience with copper-bearing lead is that the copper has an effect strikingly similar to the effect of 1 per cent antimony shown by Mr. Phillips in Fig. 6. At stresses above 400 lb. per sq. in. the creep resistance is increased, whereas below that stress it is decreased.

Mr. Phillips has compared chemical lead, Fig. 4, with an alloy of common lead and 0.065 per cent copper, in the "as rolled" condition. Conclusion 6 is based upon this comparison. If, however, these two metals are compared in the "annealed" state, no marked difference is observed in their creep resistances, according to Mr. Phillips' test results. The relative merits of these two kinds of lead seem, therefore, to depend upon the thermal and mechanical treatments to which they are subjected.

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Mr. Phillips and the American Smelting and Refining Co. are to be congratulated on the presentation of the results of these extensive series of tests, and it is to be hoped that further reports of test results will be forthcoming in the future.

MR. W. A. COWAN[2] (*presented in written form*).—H. F. Moore states[3] that creep is to be distinguished from the deformation which takes place in ordinary metals of construction, when the elastic range is exceeded.

While others[4] have defined creep without reference to whether the applied stress is above or below the elastic limit, proportional limit, or yield point, still it seems more satisfactory to the writer to agree with Moore in considering creep as having to do with phenomena occurring only at loads below the elastic limit, proportional limit or yield point (as shown by stress-strain diagrams obtained from regular tension tests). It is unnecessary to establish any exact limit as to the amount of stress indicated, particularly with metals like lead, where no such point can be definitely located. However, a limiting value can readily be taken above which creep data would be of no practical value. This may be illustrated by the curves in Fig. 1 in Mr. Phillips' paper, where the one at a stress of 550 lb. per sq. in. may be said to be in the latter category, whereas the curves at 390 lb. per sq. in. and below do represent "creep."

The terms "creep" and "creep strength" have come into use as applied to a phenomenon found to occur, but which is not indicated by the form of a stress-strain diagram representing regular tension tests. Therefore, in defining the term "creep," it should be satisfactory to limit it to the phenomenon to cover which the term was coined or applied. At higher loads, metals may flow or "run," but not "creep."

In Mr. Phillips' paper the data which are of practical value in connection with creep (excepting for some quenched samples) are all fully represented, in the diagrams, within the rectangle bounded by the vertical lines representing 10 to 1000 days, and by the horizontal lines representing 200 to 600 lb. By using this part of the diagrams only, it would enable the curves representing interesting data to be drawn on a larger scale and there would be no need of a log-scale for the stress axis, for the purpose of condensation to a diagram of convenient size. The balance of the diagrams do not represent creep, as defined above, and can well be disregarded, at least from a practical viewpoint. While the author has not shown data for stress below 200 lb., the most interesting part of the creep values, for some of the metals investigated, would apparently be found below this stress.

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[2] Assistant Chemist, National Lead Co., Brooklyn, N. Y.

[3] H. F. Moore and N. J. Alleman, "The Creep of Lead and Lead Alloys Used for Cable Sheathing," University of Illinois *Bulletin No. 48*, Vol. XXIX, February 12, 1932.

[4] R. G. Sturm, C. Dumont and F. M. Howell, "A Method of Analyzing Creep Data," *Journal of Applied Mechanics*, Vol. 3, No. 2, June, 1936, p. A-62.

Referring to Fig. 5, one of the curves shows by extrapolation a marked change in slope by a turn to the right followed by a horizontal line. This appears to be warranted by the author from his observation that no appreciable stretch was found within 100 days in one of his specimens (indicated by calipers, with an accuracy of $\frac{1}{8}$ of 1 per cent). With none of the other diagrams has a line approaching horizontal, indicating what might be called a "creep limit," been found. Apparently, such a characteristic would be found for some of the other curves within 1000 days if the stress were carried to a sufficiently low figure.

The author states that the use of log-scales for both axes has the advantage that extrapolations can be clearly indicated and their significance readily recognized. The meaning of this sentence seems somewhat obscure, particularly as to the advantage of using log-scales for both axes. It is clear that extrapolations can be indicated for the curves representing load required for time in days for 1 per cent extension, from figures obtained by extrapolating the data obtained by test, as plotted on extension-time curves. This can be done irrespective of whether or not double-log-scale diagrams are used. Presumably, in the statement referred to, the author maintains that the significance of the curve (extrapolated or not) can be readily recognized partly because of the use of log-scales for both axes. Mathematically, a straight line on the double-log-scales has a formula as follows:

$$\log y = A \log x + B$$

For a curve to the right A will represent an increasing function, and for a curve downward A will represent a decreasing function. The physical interpretation is that a straight line will indicate a certain decrease (according to the above logarithmic formula) in the rate of extension, with decreasing amounts of stress. A curve to the right indicates a greater decrease in rate of extension, while a turn downward indicates less amount of decrease in extension, with decrease in stress. A further interpretation is, that for the last two cases, the former may indicate something as to "creep limit," whereas the latter is more obscure. If no curve to the right is found, it indicates that for the lowest stress shown no supposed "creep limit" has yet been reached. In the diagrams, a curve to the right is shown only in Fig. 5. Undoubtedly, all of the curves with a downward turn must show a reversal of curvature at a somewhat lower stress if there is any approach to such a thing as "creep limit."

Without the extrapolated data from extension-time curves, extrapolation from the solid line of the curves in the stress-time diagrams is not warranted. For instance, there is no indication, from the slope of the solid line in the curves, that in Fig. 5 there will follow a sharp bend to the right, or in Fig. 4 that the lines would continue straight, and particularly in Fig. 8 nothing can be foretold as to the continuation of the downward slope of the curve for annealed material, although it is certain that at some lower stress these will show a reversal of curvature.

MR. C. R. INCE[5] (*by letter*).—In Fig. 4 of the paper, the author shows quite a variation in the creep of chemical lead between the annealed and as-rolled condition. We have found that chemical lead containing 0.06 per cent copper is an extremely stable metal and at the mild anneal used (100 C. for 1 hr.) have not found differences in properties of the proportions indicated. The differences shown by the author in this diagram may be due, in part, to the inaccuracies of the method of measuring, and it is possible that if a series of tests on chemical lead was run, the two curves plotted would fall in a shaded area similar to the one which he has plotted on common lead, but higher on every abscissa.

All our measurements on creep are made by an extensometer read by a microscope with an Ames gage attachment.[6] By using 8-in. specimens we are able to read to 0.000125 in. per inch. The variation in cross-section of specimens used by the author is not, in our opinion, the best procedure. We have found that creep rates at a particular stress and for a given kind of lead cannot be directly compared when specimens of 0.250 sq. in. and 0.750 sq. in. are used.

In Fig. 5 the author shows a series of arrows along the right-hand margin which indicate specimens that are still under test, and which have not extended 1 per cent in a period of 1000 days. Presumably the circles with arrows refer to the upper curve, common lead plus copper annealed, and the arrows alone represent common lead plus copper rolled. In both cases the author has extrapolated to the upper one of these markings. It would be of interest to know why the upper one was chosen rather than an intermediate one, for it is evidently upon the basis of this extrapolation, when compared with the chemical lead in the diagram above, that the author feels justified in drawing his conclusion that the addition of 0.065 per cent copper to a common lead produces an alloy which is superior in creep resistance.

It would seem that the validity of the author's extrapolations in Figs. 4 and 5 depends upon the meaning of these points represented by arrows. In Fig. 4 apparently he attaches more importance to preserving the general trend of his curve than to striking through his arrowed points, which is quite proper if the arrows merely signify less than 1 per cent stretch in 1000 days. In Fig. 5 he seemingly has attached more importance to passing his curve through the arrowed points than to preserving the general trends of his curves, which would appear logical only if the arrowed points signify no creep between 200 and 400 lb. per sq. in.

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[5] Assistant Sales Manager, St. Joseph Lead Co., New York City.

[6] This apparatus is described by H. F. Moore and N. J. Alleman, "The Creep of Lead and Lead Alloys Used for Cable Sheathing," University of Illinois *Bulletin No. 48*, Vol. XXIX, February 12, 1932.

A similar apparent inconsistency may be noted in Fig. 8. Here the general trend of the curve has been preserved for the lead-tin alloy in the quenched and age-hardened state in spite of two contradictory points in the 375 to 415 lb. per sq. in. range, while for the material in the as-rolled condition, more importance has been attached to the position of the point at about 210 lb. per. sq. in. than to the shape of the curve. On the basis of this curve, indications are that a stress of a little over 200 lb. per sq. in. would produce a 1 per cent extension in the same length of time as a stress of 300 lb. per sq. in. and if the curve is extrapolated further it would also appear that both a 100 and a 150 lb. per sq. in. stress would also take the same length of time.

Our own laboratory tests on tensile strength, hardness, and creep of common lead (bismuth free), do not indicate, as the author apparently believes, that copper is entirely responsible for its superiority in physical properties, to ordinary common lead. We have found hardness and tensile strength more markedly affected by addition of silver than by copper, while cobalt-nickel as well as copper, increases hardness, tensile strength, and creep resistance when added to corroding lead (Doe Run), in quantities comparable to those existing in chemical lead.

As regards creep, our results indicate that bismuth in the amounts found in present-day common leads is either negative or harmful. This may be shown by the following data on laboratory rolled specimens. Lead A is a lead over 99.98 per cent pure, in which copper and bismuth together are less than 0.0025 per cent. Lead B is a desilverized lead containing 0.11 per cent bismuth. Lead C is chemical lead containing 0.06 per cent copper and no bismuth.

	CREEP RATE, PER CENT PER HOUR		
STRESS, LB. PER SQ. IN.	LEAD A	LEAD B	LEAD C
200	5.0×10^{-5}	8.0×10^{-5}	0.4×10^{-5}
300	36×10^{-5}	48×10^{-5}	1.3×10^{-5}
400	110×10^{-5}	120×10^{-5}	3.0×10^{-5}
600	750×10^{-5}	350×10^{-5}	13×10^{-5}
800	....	....	40×10^{-5}

Bismuth-bearing leads varying from 0.005 per cent bismuth up to 0.15 per cent, with and without the addition of 0.06 per cent copper, have been investigated. The presence of 0.06 per cent copper in a bismuth lead tends to nullify any harmful effects of bismuth, as is shown by the following data, obtained from a lead containing 0.02 per cent bismuth to which 0.06 per cent copper has been added:

STRESS, LB. PER SQ. IN.	CREEP RATE, PER CENT PER HOUR
200	2.8×10^{-5}
300	6.5×10^{-5}
400	21×10^{-5}
600	100×10^{-5}
800	980×10^{-5}

From the above data it seems evident that, whereas the addition of the 0.06 per cent copper has probably improved the creep resistance of the bismuth lead, it is still inferior in this property to the straight chemical lead containing 0.06 per cent copper and no bismuth.

MR. ALBERT J. PHILLIPS[7] (*author's closure by letter*).—Mr. Betty has pointed out quite correctly that creep tests may be designed for different purposes, and, consequently, reveal different results. The tests reported in the paper were all designed to compare different commercial leads and alloys, using a standardized technique in their preparation, so that the results are strictly comparable for that particular technique. I believe that in view of the differences in materials tested, the agreement of the work reported in this paper with that reported by the University of Illinois, is quite satisfactory. Mr. Betty calls attention to the fact that the 0.065 per cent copper-lead compared in the as-rolled condition with chemical lead, showed different results, whereas in the as-annealed condition, showed approximately the same results, and states that the properties of these two leads depend upon the thermal and mechanical treatments. This conclusion is correct; we have been able to check these results with several different specimens. It is our opinion that there may be residual strain in chemical lead in the as-rolled condition, which is, usually, the normal commercial condition, and that such residual strains may be responsible for the greater degree of creep. In the as-annealed condition, the important factor in controlling creep seems to be merely the copper content.

I cannot agree with Mr. Cowan's differentiation between creep and flow, or run. In fact, I prefer to consider a creep test as being quite similar to a tension test, differing mainly in the factor, time. Limiting the definition of creep to loads below the elastic limit, proportional limit, or yield point, does not seem at all practical, for the determination of these points depends almost entirely upon the speed of testing. It might be possible to define creep as flow occurring below the instantaneous elastic limit, but the determination of the instantaneous elastic limit would be, at best, an extremely controversial matter. The high loads were shown in the creep diagrams (given in the paper) for a specific purpose, namely, to emphasize the importance of speed in tension testing, and to emphasize still further the fact that short-cut creep tests, whereby comparisons are made of rates of creep at a constant load, may be, and usually are, thoroughly misleading. There are many papers in the literature presenting so-called "creep data" on lead, in which conclusions are drawn from the rates of creep of different alloys under a fixed load of 500 lb. per sq. in. The data given in the paper are useful in showing the fallacy of such methods of testing. In addition, the advantage of reporting the high load values lies in the fact that an experienced metallurgist will recognize such values as a partial definition of the condition in which the metal was tested initially and makes it possible for him to compare the condition of the creep specimens with the condition of leads prepared by other methods.

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[7] Superintendent of Research, Research Dept., The American Smelting and Refining Co., Maurer, N. J.

With regard to plotting values below 200 lb. per sq. in., it should be borne in mind that the purpose of the research was to develop alloys of superior creep strength; and, consequently, any alloy which showed rapid creep at 200 lb. per sq. in. is of no interest to those searching for leads with superior creep resistance. I quite agree with Mr. Cowan that other alloys may have a limiting creep stress, as indicated by a horizontal line in the diagrams, if the loads were carried low enough, and, in fact, we have later data from tests that have been in progress for a little less than two years to support this contention.

Mr. Cowan's objections to the use of a log scale for the stress axis seem to be based mainly upon a desire to determine the mathematical formula for the creep curves, which, presumably, would permit extrapolation of creep rates. We have carefully avoided extrapolating creep rates and feel that there is no justification at the present time for extrapolating or for a mathematical analysis of the creep data. The main advantage in using a log scale on the stress axis permits plotting low stresses, where variations are important, with a wide scale and recording on the same graph the high stresses to indicate the condition of the metal or alloy. The author does not feel that there is sufficient data at the present time to attach significance to change in slope of curves, with the possible exception of changes in slope to approach a horizontal asymptote indicating creep limit. It should be remembered that the mechanism of creep may be very complex, particularly with alloys, involving residual strain, recrystallization, precipitation hardening, particle size, grain growth, boundary movements, and other phenomena.

Mr. Ince's comment that chemical lead is extremely stable and is not influenced by a mild anneal at 100 C. for 1 hr. is not borne out by many tests made in this laboratory. Our tests show consistently that chemical lead, as-rolled, has different properties from those obtained after annealing for 1 hr. at 100 C. However, a carefully standardized testing technique has to be employed in order to eliminate variables which cause differences greater than those obtained by the anneal. The differences plotted in our diagrams are not due to inaccuracies in the method of measuring but are due to true differences in the specimens. These differences can be shown by repeated tests involving much more accurate methods than those indicated in the paper, and even more accurate methods than those reported by Mr. Ince. Our present method of determining strain involves the use of a portable microscope and a calibrated gage length mounted on the specimen. We are able to determine accurately changes of 0.0001 in.

in 10 in., which is equivalent to 0.00001 in. per inch, more than ten times the accuracy reported by Mr. Ince. Tests with this modified equipment prove quite conclusively that the differences given in the paper are real and represent differences in the alloys. We have found that creep does not progress continuously but intermittently, and in some cases, rest periods with practically no flow for as much as 200 days, have been encountered. These tests were all conducted in a constant-temperature room, held at 30 C., plus or minus $\frac{1}{4}$ of a degree. While we have adopted the use of delicate strain measurements, our data indicates that extrapolations can be off as much as 1000 per cent, and that the only method of determining creep rates that can be safely used, is the long-time method.

Mr. Ince's statement to the effect that the variation in cross-section of specimens employed by the author is not good practice needs considerably more support than he has advanced in the discussion. It should be noted that all specimens of any series were made from rolled strip having exactly the same thickness. The only variation was in the width of the strip, from a minimum of $\frac{1}{2}$ in. to a maximum of $1\frac{1}{2}$ in. The variation in results due to this variation in width is extremely small as compared to other variations involving the fundamental properties of the lead being tested, particularly those due to residual strain, grain growth and precipitation phenomena.

Mr. Ince's comments with regard to the extrapolation of the curves in Fig. 5 are a little difficult to understand. It is quite obvious that if no one of a group of specimens having progressively higher loads has extended 1 per cent, then the curve for 1 per cent extension should go through a point representing a load higher than any one of the specimens being tested. Mr. Ince's contention that the curve might just as well go through an intermediate point, does not seem logical. The extrapolations in both Figs. 4 and 5 were all based upon the relative creep of the specimens. While none of the specimens indicated by the arrows had extended 1 per cent, several of them had extended appreciable amounts, approaching 1 per cent, and consequently, if there is any justification for extrapolating, the curves, as shown, can be defended. The author has made extrapolations in the paper only where specimens have been under test and have failed to extend 1 per cent. No extrapolations have been made to times beyond the test conducted. Quite obviously, such extrapolations are very conservative.

Mr. Ince states that the author believes that copper is entirely responsible for the "superior" physical properties of chemical lead. The author has not stated that chemical lead is superior in physical properties to ordinary common lead, and certainly could not imply that copper is entirely responsible for such superiority. The author agrees that chemical lead has a higher tensile strength than common lead. This increase in strength lies entirely in the fact that chemical lead is not pure lead but contains approximately 0.06 per cent copper, 2 oz. of silver per ton, as well as minor amounts of nickel and cadmium. All of these elements are not contained in common lead, in other than minute amounts in the fourth decimal place. The copper is very largely responsible for stiffening chemical lead as compared to common lead, but it can be demonstrated very readily with simple tension tests that the silver, and possibly nickel, likewise stiffen the lead. We have not been able to demonstrate that the addition of silver to lead, or that the presence of silver in chemical lead effects any improvements whatever in creep resistance. We have added silver to pure lead, to copper-bearing leads, and to antimonial leads, and in no case have we obtained improved creep resistance, but in several cases lower creep resistance has resulted. On careful investigation, Mr. Ince will find that whereas the addition of 2 oz. of silver per ton to pure lead does produce a considerable increase in hardness and tensile strength, it does not produce a commensurate increase in hardness and tensile strength in a copper-bearing lead; and nickel added to a copper-bearing lead affects the tensile strength and hardness by less than 1 per cent.

With regard to Mr. Ince's data on creep, it would be of interest to know the details of his tests, such as the size of specimens, the preparation, the composition (other than the nominal analysis), the method of testing, and particularly, the time of testing. He does not state whether or not his creep rates were determined on 30-day tests, 90-day tests, or 3-yr. tests, such as have been reported by the author. It is obvious that a comparison of data obtained on, for example, a 30-day test with those obtained from 3-yr. tests, is quite inadequate and may be misleading. Mr. Ince states first that bismuth added to lead has either a negative effect or lowers the creep resistance. We will agree with Mr. Ince that large amounts of bismuth added to lead definitely lowers the creep resistance, but these amounts must be several per cent. We have data on alloys containing 1, 3 and 20 per cent of bismuth. The 20 per cent bismuth alloy creeps at an extremely high rate; the 3 per cent bismuth alloy creeps at a rate appreciably higher than that for pure lead; the 1 per cent bismuth alloy probably creeps at a rate greater than that for pure lead but the difference is rather small, and if it were not known that greater amounts of bismuth accelerate creep, the author would not feel sure that even 1 per cent bismuth has an appreciable effect. It is felt that the effect of adding 0.1 per cent bismuth to lead is below the sensitivity of the most delicate creep technique devised to date. The author is quite sure that if Mr. Ince will compare his data on leads A and B with creep data on other tests on pure and common leads, he will find that the agreement is quite within the reproducibility of his tests.

With regard to his data on lead C, the rates of extension at 400, 600 and

800 lb. per sq. in. are definitely lower than we have experienced for chemical lead or any other copper-lead at these loads. At loads of 300 and 200 lb. per sq. in., we do not have comparable data, for at the low loads the rate of extension is so low that in order to secure 1 per cent extension, according to Mr. Ince's own data, it would take $28\frac{1}{2}$ yr. at 200 lb. per sq. in. The author feels that the accuracy of extrapolating on very much less than 1 per cent extension is open to serious question. Consequently, it is not felt that Mr. Ince is justified in comparing what are obviously rates based on minute extensions with rates which have been determined, on extensions of 1 per cent, and tests of now four years' duration. All of our tests on copper-bearing lead, regardless of whether it is chemical lead, or compounded copper-lead, made from either special high-purity lead (99.999 per cent lead) or common lead, show rates of extension at least as low as those given by Mr. Ince for chemical lead at 200 lb. per sq. in. For example, a specimen produced from high-purity lead by the addition of 0.065 per cent copper loaded at 213 lb. per sq. in., has shown a rate of extension over a period of 372 days of less than 2.2×10^{-7} per cent per hour, a value several hundred times lower than that reported by Mr. Ince for chemical lead. However, this same lead loaded to 347 lb. per sq. in. showed a creep rate of 10×10^{-5} per cent per hour, or a value more than three times as large as Mr. Ince gives for chemical lead at 400 lb. per sq. in. These samples were prepared identically, suspended at the same time in the same rack, in the same constant-temperature room for 372 days. At the same time that these specimens were started, additional specimens, prepared identically, but with the additions of 0.006 per cent silver and 0.004 per cent nickel, thus duplicating for all practical purposes the analysis of chemical lead, were started. At the end of a year, a specimen of this synthetic chemical lead, loaded to 228 lb. per sq. in., showed a creep of 1.0×10^{-5} per cent per hour, thus checking reasonably well Mr. Ince's figures. However, the author does not feel that such comparisons are at all fair, for they are based on extremely minute extensions—in the case of the last specimen mentioned, extensions of about 0.005 in., while in the preceeding case at the low load, extensions of 0.0002 in. Such extensions can be very largely influenced by minor inequalities in the grips and even by small bends in the test specimens to say nothing of strain adjustments in the specimens. In this connection, it should be noted that the present procedure used by the author involves full swivel grips at both top and bottom of the specimen with positive center alignment, so that the inequalities due to non-axial suspension have been reduced to the lowest possible minimum.

The figures given by Mr. Ince for creep of leads containing 0.02 per cent bismuth and 0.06 per cent copper show much poorer creep resistance than we have ever obtained on such an alloy over a series of at least two dozen sets of tests. Mr. Ince does not give the complete analysis of the samples which he tested, and this is very important, for while we do not feel that minute amounts of most impurities affect the creep resistance of pure lead, we do feel that certain impurities can combine with copper, for example zinc and tin, practically nullifying the effect of the addition of copper to lead. This may account for the results which Mr. Ince has experienced. The author feels that until Mr. Ince has stated much more exactly his composition, technique, and particularly the duration of his tests, his data cannot be accepted unconditionally.

THE EFFECT OF THE ADDITION OF LEAD ON THE ENDURANCE LIMIT OF A CERTAIN TIN-BASE BEARING ALLOY

BY JOHN N. KENYON[1]

SYNOPSIS

Continuation of an investigation started several years ago to determine the effect of the addition of lead on certain tin-antimony-copper babbitt metals (**4**)[2] shows that the addition of 4 per cent of lead has little effect on the endurance properties of a tin-base bearing alloy (80 per cent tin, 15 per cent antimony, 5 per cent copper), and that if lead has any effect, it is to improve the material. It has also been discovered that a peculiar form of bearing failure occurs after the alloy has been in service a long time, and some explanation for this failure is offered.

American practice strictly limits the lead content of tin-base babbitt metals because of the supposed softening and embrittling effects. On the other hand, European practice often permits the addition of small amounts of lead, even when these alloys are to be subjected to severe service such as in Diesel engine crankshaft bearings. The main bearings of American automobile engines, for instance, are lined with tin-base babbitt metal about 0.031 in. ($\frac{1}{32}$ in.) thick, and the lining may absorb lead from the "tinning" operation. This absorption of lead may justify, to some extent, a low lead tolerance in the standard specifications. It is yet to be determined, however, whether lead should be considered as an injurious impurity or a valuable alloying element, and it is hoped that this study may throw some light on the question.

A Brief Survey of Other Recent Findings:

Ellis and Karelitz (1928–1930) found that lead adds something to the mechanical strength of a tin-base bearing alloy and has an important effect on the microstructure. It was suggested that "the microstructural changes occasioned by lead tend to confirm the current opinions regarding its embrittling action"(**2**).[2] They found that "wiping" may occur at temperatures of 170 to 180 C. due to the presence of a tin-lead eutectic even though the lead content is within the tolerance set by American practice (0.34 per cent) (**3**).

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[1] Testing Engineer, Civil Engineering Testing Laboratories, Columbia University, New York City.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 197.

Cowan (1928) found some tin-base bearing alloys "to contain shrinkage cavities when small percentages of lead are present, but to be practically free from similar cavities with no lead content" (1).

Kenyon (1933) found that the addition of 3 to 4 per cent of lead to some tin-base bearing alloys increased the hardness of these materials for temperatures up to 100 C. No important falling off in hardness was found for temperatures up to the melting point of the tin-lead eutectic (183 C.) but at higher temperatures the alloys containing over 2 per cent of lead showed a tendency to crumble under the Brinell load (4).

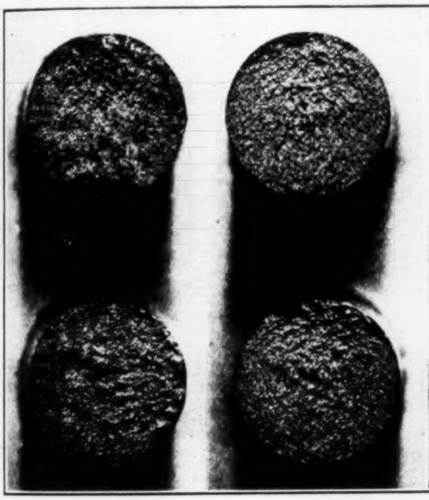

FIG. 1.—Fatigue Fractures Obtained in Sound Metal.

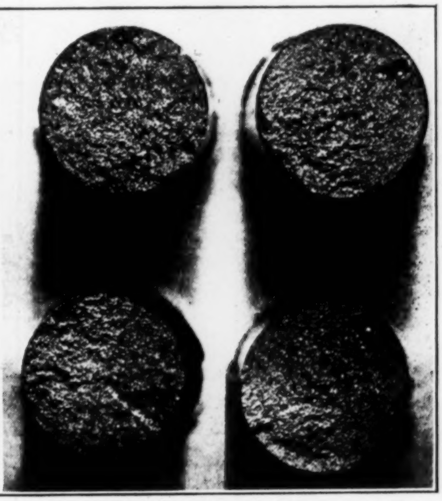

FIG. 2.—Fatigue Fractures Due to Defects. The lower left fracture was caused by a microscopic surface inclusion; the other three are attributable to gas cavities.

MacNaughtan (1934) discusses the general question of white bearing metal service failures and draws the following conclusion:

"Although failure by pounding is occasionally important, the outstanding difficulty experienced in current severe conditions, particularly internal-combustion engines, is a peculiar form of fatigue cracking. After a period of service under pulsating stress and high temperature, numbers of fine cracks appear on the surface of the bearing and spread in all directions to form a network similar in appearance to a tesselated pavement." MacNaughtan continues: "A considerable amount of data has been obtained (on white metals) with respect to wear resistance, friction under test conditions in bearings, and such physical properties as are revealed by determinations of Brinell hardness, proportional limit in compression, etc. There is, however, almost an entire absence of information concerning fatigue properties" (5).

Method of Test:

A babbitt metal (80 per cent tin, 15 per cent antimony, 5 per cent copper) was prepared from commercially pure materials and to one-half of this alloy 4 per cent of lead was added. These two batches of metal were then cast in triangular-shaped solder molds ($\frac{5}{8}$ in. deep and $\frac{7}{8}$ in. wide at top) so as to avoid any chance of segregation. The material was carefully remelted and chill cast at 700 F. (370 C.) into $\frac{1}{2}$-in. diameter rods about 5 in. long. These rods were annealed at 212 F. (100 C.) for 12 hr. to remove casting stresses and were then machined into standard R. R. Moore fatigue coupons. The test specimens were given as good a polish as possible on the lathe and a final longitudinal polish, using No. 000 Hubert paper. The fatigue machines were operated at a speed of 3750 r.p.m., with bearing temperatures of about 140 F. (60 C.). The temperature assumed by the test coupon was very nearly this value.

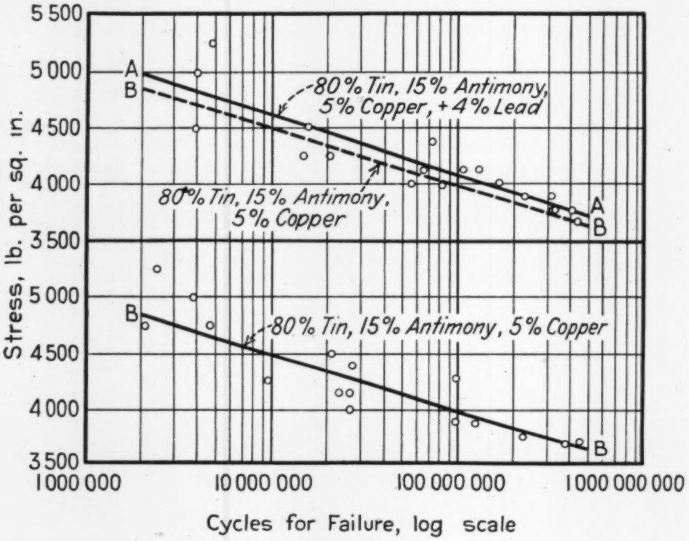

FIG. 3.—Summary of Results.

Discussion of Results:

The appearance of the fatigue fractures is shown in Figs. 1 and 2. The fractures are similar to fatigue breaks obtained on the harder "elastic" materials wherein a smooth crack spreads over a large section of the specimen before final fracture. Careful measurements of the broken test coupons showed no evidence of "creep," and it is believed that rate of loading did not affect results.

Figure 1 shows the appearance of fractures obtained in sound metal and Fig. 2 the appearance of fractures attributable to defects. Three of these breaks were caused by gas cavities, and the fourth by a microscopic surface inclusion not clearly discernible in the photograph. The defective specimens failed shortly after being placed in the fatigue machine.

Figure 3 is a summary of the results of the tests. The addition of lead appears to have little effect on the endurance properties of the alloy except possibly to improve it. There is nothing in the results to justify the current opinion that lead has an embrittling effect on the alloy (2). The endurance properties of the alloy do not appear to be impaired by the addition of lead as would be expected if minute shrinkage cavities occurred (1).

The trend of the graphs would seem to indicate that there is no *definite* endurance limit, and ultimate failure may occur at lower stresses after a considerably greater number of repetitions. To some extent this deduction would explain the "peculiar form of fatigue cracking" mentioned by MacNaughtan (5).

A period of three months is required for subjecting one test coupon to 500,000,000 stress reversals, and time did not permit carrying the investigation any further.

Acknowledgments.—Appreciation is due Prof. William Campbell and Prof. James K. Finch for their encouragement.

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(1) W. A. Cowan, "Minute Shrinkage Cavities in Some Cast Alloys of Heterogeneous Structure," *Journal*, Inst. Metals, Vol. 39, No. 1, p. 53 (1928).

(2) O. W. Ellis and G. B. Karelitz, "A Study of Tin-Base Bearing Metals," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 49–50, Part II, p. MSP-50-11 (1927–1928).

(3) O. W. Ellis and G. B. Karelitz, "A Study of Tin-Base Bearing Alloys," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 52, Part II, p. MSP-52-7 (1930).

(4) J. N. Kenyon, "The Effect of the Addition of Lead on the Hardness of Certain Tin-Base Bearing Alloys at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 430 (1933).

(5) D. J. MacNaughtan, "The Improvement of White Bearing Metals for Severe Service," *Journal*, Inst. Metals, Vol. 55, No. 2, p. 33 (1934).

DISCUSSION

Mr. G. B. Karelitz[1] (*presented in written form*).—In spite of the fact that various specifications definitely require lead-free tin-base babbitt, lead is always present in bearing linings. It appears in small amounts in commercial tin. The Straits tin was found to be practically free of lead, while the commercial "99 per cent" tin contains up to 1 per cent of lead. Furthermore, it is the general practice to tin the bearing shells prior to pouring the lining with 50–50 or rather 60–40 lead-tin solder. The total amount of metal poured into the shell is small, particularly with centrifugal casting of bearing linings, and the lead content of the actual lining may reach several per cent.

The writer found, while working on tin-base bearing metals with Mr. O. W. Ellis, that the compressive strength and Brinell hardness of the metal increased with the addition of up to 1 per cent of lead. Further addition of lead, up to 3 per cent did not produce any effect. At elevated temperatures, the effect of lead on the strength of the metal was negligible. It was our impression at the time that the effect of lead could be explained satisfactorily by the formation of massive needles of copper-tin in the presence of lead. However, it must be realized that the tests were performed on metal cast in cylinders, while the behavior of babbitt metals in a thin lining is different from that in a standard test specimen.

The information submitted by Mr. Kenyon on fatigue properties of tin-base babbitt metals is of definite value. The temperature of the test specimens, near 140 F., was fortunately in the region of normal bearing temperatures. It is worth while noting, however, that the cracking failure mentioned in the paper takes place apparently when the bearings run at a much higher temperature, this may reach nearly 300 F. in automobile or Diesel engines. The writer is tempted to suggest extending the fatigue tests to specimens in the shape of linings, cast, say, onto thin steel or bronze plates, and stressed at elevated temperatures. It would be important to decide whether cracking of the lining is due to mechanical or thermal causes.

Mr. H. F. Moore.[2]—Mr. Kenyon's statement that during his tests no creep phenomena were observed, is doubtless correct. However, it may be possible that a slight amount of creep took place in the lead-containing part of the bearing alloy—an amount so small that it produced no appreciable effect on the specimen as a whole but which might have served to start spreading cracks in the lead-containing parts of the metal.

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[1] Associate Professor, Department of Mechanical Engineering, Columbia University, New York City.
[2] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

For bearing metals fatigue tests in *repeated compression* would seem promising for giving results which might serve as an index of bearing strength. Bearing metal is frequently subjected to repeated compression, and experiments on other metals under repeated compression show that fatigue cracks can be started under cycles of repeated compression, probably due to the shearing stresses set up on diagonal planes.

MR. C. E. SWARTZ.[3]—The statement made by Mr. Karelitz that bearing metal is bonded with 50–50 or 60–40 lead-tin alloy is true in some bearings not of the precision type, such as ordinary line shafting, or cast connecting rod bearings. However, so far as the automotive industry is concerned, cases of this type are rare. The great majority of the bearings in use are bonded with pure Straits tin of the very highest quality that can be purchased. Most of the severe bearing services today are at rather elevated temperatures. In the laboratory of the Cleveland Graphite Bronze Co. we are making fatigue tests at 300 F. We are finding that test to be somewhat more severe than Mr. Kenyon's test. The tests do not run long; many may be made in a short space of time. In addition, data at this high temperature have been found to be much more significant than when run at room temperature. As to the influence of lead, while we have no definite information, the influence is not marked at that high temperature except when it is in excessively high amounts. However, all bearing materials are held to a minimum of lead, never over 0.35 per cent.

MR. R. L. TEMPLIN.[4]—I should like to ask the author whether, in his studies, particular attention was paid to grain size in the specimens on which fatigue tests were made. Usually in specimens of this type there is a marked difference in fatigue properties, depending on grain size.

MR. JOHN N. KENYON.[5]—Replying to Mr. Karelitz, endurance tests made on cast linings held at elevated temperatures would probably give interesting data since these tests more nearly simulate service conditions. However, it too often happens that mechanical difficulties are encountered when we depart from the standardized test and these studies are too time-consuming for lengthy preliminary experimentation. The study would naturally involve the question of using a pure tin or tin-lead for the "tinning" operation.

I believe Mr. Moore's statements are essentially correct. An entire absence of "creep" cannot be expected to be found in these materials. The point I want to make is that the fatigue fractures were more characteristic of the harder elastic materials than of a softer material, such as lead, in which the phenomenon of creep is pronounced.

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[3] Metallurgist, Cleveland Graphite Bronze Co., Cleveland, Ohio.
[4] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.
[5] Testing Engineer, Civil Engineering Testing Laboratories, Columbia University, New York City.

In reply to Mr. Templin, we have not made tests to determine the effect of grain size in that this study was undertaken primarily to determine the effect of lead on the endurance limit. The two materials were cast under identical conditions and any difference in structure may be directly attributed to the lead addition.

EFFECT OF IRON IMPURITIES ON THE ANNEALING OF HIGH BRASS

By W. A. Gibson[1] and J. H. Doss[1]

Synopsis

This paper gives the effect of iron impurities on the annealing of high brass for temperatures from 600 to 1200 F. and for iron impurities from 0.008 to 0.287 per cent. It pays particular attention to the effect of annealing at temperatures from 800 to 900 F. and for iron impurities below 0.10 per cent. There is also indicated the effect of time on the annealing of brass. It is in the detailed attention paid to the lower iron contents and to the lower annealing temperatures that this paper differs from previously published information. By such attention information is brought out which is not noticeable with less-detailed data.

Introduction

In the brass-rolling mills it has long been known that varying amounts of iron in high or cartridge brass sheet cause erratic annealing. As a result an upper limit of 0.05 per cent iron is customarily set for quality brass such as that used for cartridge cases. For other uses the maximum limit is variously set from 0.05 to 0.10 per cent. The great increase in the use of polished and chromium-plated parts, especially on automobiles, in the last 5 yr. has made necessary a very close control of grain sizes and tempers. Grain-size limits of ± 0.005 mm. are now the rule in much sheet brass shipped in strips. The effect of increased iron is to retard grain growth. Therefore, variations of iron impurity make close control of grain size difficult. Little, if any, quantitative data have been published on the subject.

Cook and Miller[2] published the results of an investigation on cartridge brass. Although the material used differed from that of the present investigation the results are sufficiently similar to make a short review of their data worth while. Figure 1 is a reproduction of one of the figures of their paper. This shows that the temperature of initial recrystallization is only slightly affected, if at all, by iron impurities from 0 to 0.49 per cent; that the resulting temper becomes harder as iron increases; and that there is a secondary recrystallization which begins at 500 to 600 C. (930 to 1110 F.), depending upon the iron content, and is practically completed for all iron contents at 700 C. (1290 F.)

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[1] Supervisor of Methods, and Metallurgist, respectively, Revere Copper and Brass Inc., Detroit, Mich.

[2] Maurice Cook and H. J. Miller, "The Effect of Different Elements on the Annealing and Grain Growth Characteristics of Alpha Brass," *Journal*, Inst. Metals, Vol. XLIX, No. 2, p. 250 (1932).

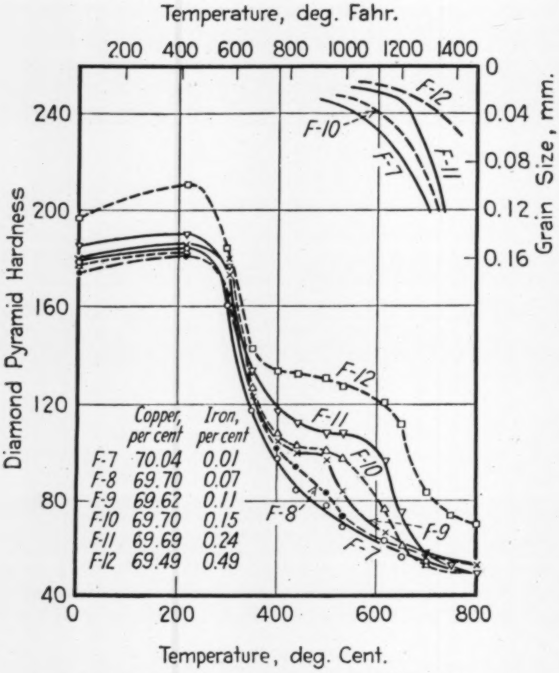

FIG. 1.—Hardness Data of Cook and Miller's Paper.

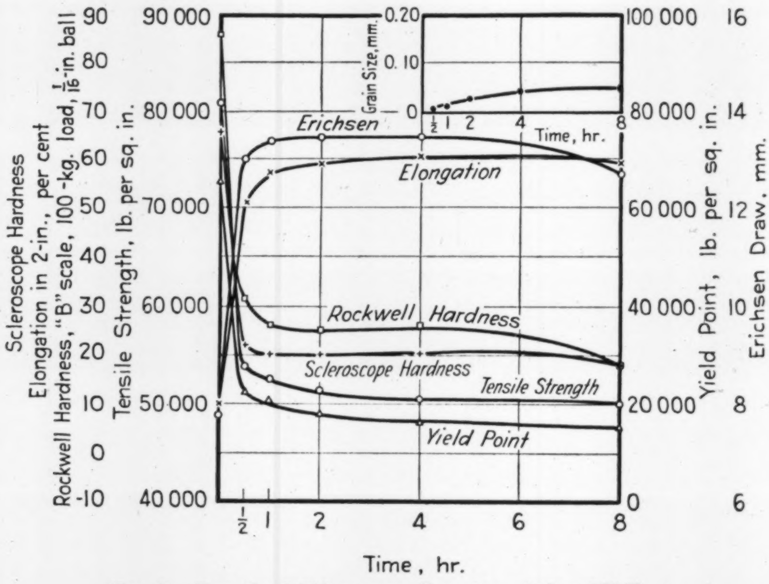

FIG. 2.—Data for 0.049 per cent Iron Annealed at 900 F.

Their work has not covered sufficient samples near and below 0.10 per cent iron to determine quantitative effects of the amounts commonly present in brass. It is this field the authors seek to cover.

PROCEDURES

Samples were obtained having the following analysis:

SAMPLE	COPPER, PER CENT	LEAD, PER CENT	IRON, PER CENT
No. 1	66.4	0.030	0.008
No. 2	65.9	0.010	0.018
No. 3	66.4	0.080	0.040
No. 4	66.7	0.030	0.049
No. 5	66.5	0.080	0.066
No. 6	66.2	0.12	0.085
No. 7	65.8	0.01	0.118
No. 8	64.7	0.01	0.140
No. 9	64.5	0.01	0.204
No. 10	64.1	0.01	0.287

These were cast into bars 6 by 2 by 65 in. and were cold rolled to 0.065 in. thick by reductions of approximately 50 per cent between anneals at 1200 F. At 0.065 in. the metal was annealed at 1050 F. for 2½ hr. and was then cold rolled to 0.040 in. thick. All the tests were carried out upon metal at this gage.

Standard A.S.T.M. tension test specimens and small squares were cut from these strips. These were then placed in an electric muffle furnace (4 by 3½ by 10 in.) automatically controlled to ±5 deg. Fahr. by a recording potentiometer. The furnace was already at the required temperature and the control thermocouple was so placed that it rested on the surface of the test specimen.

After the furnace had regained the drop in temperature, due to opening of the doors and inserting the cold charge, samples were removed at intervals of ¼, ½, 1, 2, 4, and 8 hr. This was carried out at 600, 700, 800, 900, 1000, 1100, and 1200 F., and the resulting data plotted.

The data obtained consisted of Rockwell and scleroscope hardnesses, yield point, tensile strength, elongation, Erichsen value, and grain size.

Rockwell hardness was measured on small specimens 1 in. square. These were pickled, washed and dried in sawdust. A 100-kg. load with $\frac{1}{16}$-in. ball ("B" scale) was used. The equipment was calibrated using standard test blocks purchased from the instrument manufacturer.

Scleroscope hardness was measured on the same specimen using a magnifier hammer which, on brass, gives a reading 1.75 times the standard universal hammer.

Yield points were taken with dividers at stress corresponding to 1 per cent elongation.

Tensile strengths were obtained using a 50,000-lb. beam-type testing machine and specimens prepared for a 2-in. gage length in accordance with

A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 – 33).[3]

Erichsen values were obtained using the standard equipment which has a semi-spherical punch of 10-mm. radius with a 27-mm. inside diameter hold-down ring. All samples were prepared for test in the same manner as the Rockwell specimens. Both punch and metal were entirely free of oil or any other lubricating material during the test.

Grain sizes were estimated to the closest 0.005 mm. by comparison with the chart which forms part of the A.S.T.M. Standard Rules Governing the Preparation of Micrographs of Metals and Alloys (E 2–30).[4] Observer's errors in comparison were occasionally checked by actual grain counts.

The iron analyses were made by the iron-oxide method. Three independent sets of analyses were made and were not accepted until all three sets checked.

DATA

The resulting data were plotted on seventy charts, each temperature with each iron content being plotted as one chart. Only one illustrative figure is given here which shows the plotted data for 0.049 per cent iron annealed at 900 F. for the times shown (Fig. 2).

From these plots the values obtained after two hours annealing have been taken. Rockwell values for various temperatures are as shown in Fig. 3, and the corresponding grain sizes in Fig. 4. The other physical values were obtained but those shown give sufficiently closely the effect of iron impurities.

For those interested in tensile strengths we find that in this work the following approximate relationship holds for samples annealed between 700 and 1200 F. It does not hold for hard or for imperfectly recrystallized metal:

$$T = 310\text{ B} + 43{,}000$$

where T = the tensile strength in pounds per square inch, and
B = the Rockwell hardness using a 100-kg. load with a $\frac{1}{16}$-in. ball.

DISCUSSION OF RESULTS

The figures given are self explanatory. We find that at about 0.09 per cent iron there is a very sudden change in annealing properties. This change is so sudden that great non-uniformity of product will result if brasses containing more iron than this are processed with those containing less.

On the other hand, high iron contents are not harmful and may even be beneficial in certain cases, as for example in quarter-hard material where a fine grain on the finish material is required.

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[3] 1933 Book of A.S.T.M. Standards, Part I, p. 949.
[4] *Ibid.*, p. 900.

Figure 4 shows the inversion which is indicated but is not definite in the hardness data of Cook and Miller's paper (see Fig. 1). For the higher annealing temperatures iron contents over 0.09 per cent may actually assist in recrystallization.

We suggest, as a possible explanation for the sudden discontinuity in effect of iron, the formation of a copper-iron-zinc compound at the grain boundaries during the initial recrystallization; that this enveloping boundary prevents further amalgamation of grains until it reaches a high temperature at which it is taken into solution and annealing again occurs. Such an explanation would account for the secondary recrystallization as reported by Cook and Miller, as well as the divergence, at high iron content of the curves shown in Fig. 4.

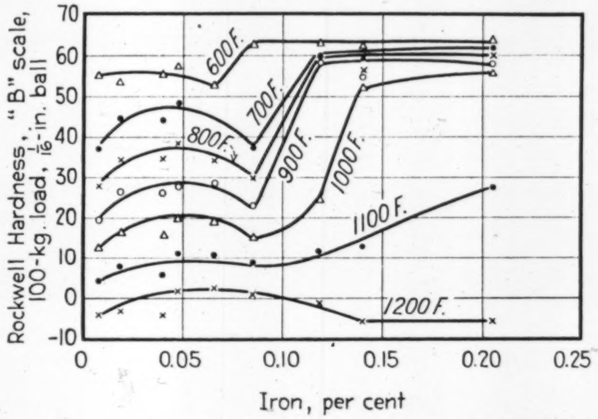

FIG. 3.—Showing Effect of Iron on Rockwell Hardness of High Brass.

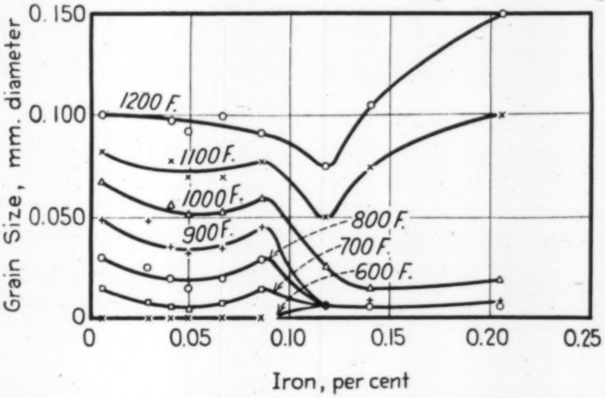

FIG. 4.—Showing Effect of Iron on Grain Size of High Brass.

That the retarding influence is not present at the start of annealing is shown by the negligible effect which iron has on both the initial recrystallization temperature and the time required for this initial recrystallization. At the lower temperature iron content in excess of 0.09 per cent very slightly reduces both the temperature and time required for the initial recrystallization.

The iron percentage at which the sudden discontinuity occurs can also be shifted by traces of other impurities, but a discussion of these other impurities is outside the scope of this paper.

CONCLUSIONS

1. In high brass having lead and iron as the only impurities, iron contents as high as 0.09 per cent are not harmful to annealing.
2. In such material a great discontinuity in annealing properties occurs between 0.09 per cent and 0.118 per cent iron.
3. The presence of iron greater than 0.118 per cent is not harmful to sheet high brass provided proper allowance is made in processing and use for its annealing properties. If these allowances are not made it is very harmful.
4. A need exists for information on the annealing properties of high brass containing less than 0.005 per cent iron.

Acknowledgments:

We desire to express our thanks to Messrs. Cook and Miller for permission to use a part of their data; to our colleagues Messrs. Stedman, Oestrike, and Hawkins who have carried out most of the tests required; and particularly to the management of the Revere Copper and Brass Inc. for permission to publish data which have been accumulated in their Michigan Division as a part of plant control.

THE FORMING PROPERTIES OF SOME NON-FERROUS SHEET METALS

By G. R. Gohn[1]

Synopsis

This paper continues an investigation which was first undertaken several years ago by the Western Electric Co. to obtain information relative to the forming of 90-deg. bends in various non-ferrous sheet metals. The previous paper[2] described a method whereby such forming data might be obtained and tabulated minimum safe forming radii for three alloys of brass and two alloys each of nickel silver and phosphor bronze. This information has proved so valuable in the design and manufacture of telephone apparatus that the investigation was extended as described in the present paper. Additional forming data are presented on the seven alloys previously studied as well as data on other non-ferrous sheet metals. The method used in determining the minimum safe forming radius is described and the data so obtained are tabulated.

Contact springs and many structural parts of telephone apparatus are made from thin sheets of non-ferrous metals in a series of blanking, punching and forming operations. Such formed metal parts may vary in size from small springs made from sheet metal only 0.005 in. thick to large cases made from $\frac{3}{8}$-in. stock, but the bulk of the sheet material used is smaller than No. 14 B. & S. gage stock.

Formed parts which are bent over too sharp a radius may crack as shown in Fig. 1, specimens Nos. 1 to 5. This cracking results from stressing the outer fibers of the metal beyond the point of rupture during the forming operation. When the overstressing is less severe, an "orange peel" effect is produced. This is illustrated in specimens Nos. 6 and 7 of Fig. 1. Such a condition, while not as objectionable as an open crack, is unsightly in finished apparatus, and because of the overstrained condition of the metal, early failure of the part may result through accidental overstressing in service or through corrosion. It is therefore desirable from a design and manufacturing standpoint to have available data relative to the forming properties of the different thicknesses and tempers of the various sheet metals used. Considerable information of this type has been presented in a previous paper[2] covering the more important materials used in the manufacture of telephone apparatus. This information has proved so helpful in the design and manufacture of telephone apparatus that the forming studies were extended to obtain additional information on the forming properties of the principal non-ferrous sheet metals previously studied as well as certain other commercial non-ferrous metals.

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

[2] W. A. Straw, M. D. Helfrick, and C. R. Fischrupp "Forming Properties of Thin Sheets of Some Non-Ferrous Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Division, p. 317 (1931).

In the paper referred to,[2] a method was described for determining the minimum safe forming radius for a given material from a series of specimens formed over 90-deg. V punches. The method consists in taking specimens approximately $\frac{3}{4}$ by 3 in. cut from a sheet of metal parallel, at 45 deg., and at 90 deg. to the direction of rolling. These specimens are then formed over each of a series of punches having various radii until a satisfactory forming radius is obtained. Whereas the punches used by the previous investigators[2] had sharp, $\frac{1}{64}$, $\frac{1}{32}$, $\frac{1}{16}$, $\frac{3}{32}$ and $\frac{1}{8}$-in. radii, it was felt desirable

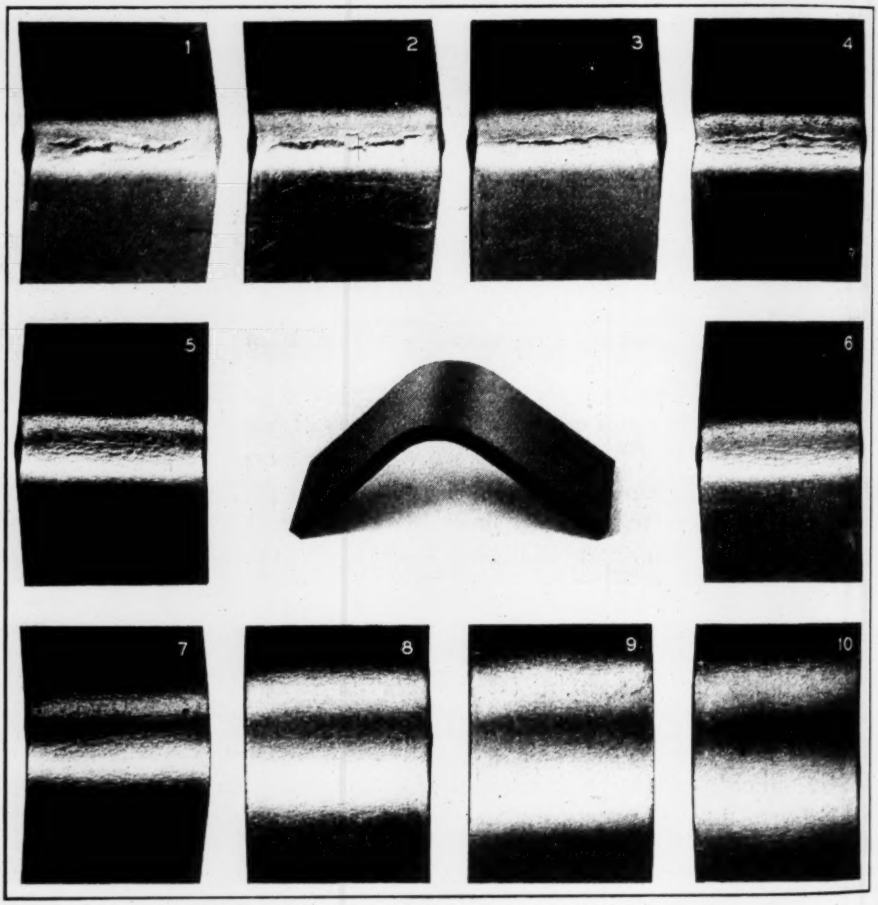


FIG. 1.—Sections of Formed Specimens (Insert—Satisfactorily Formed Specimen).

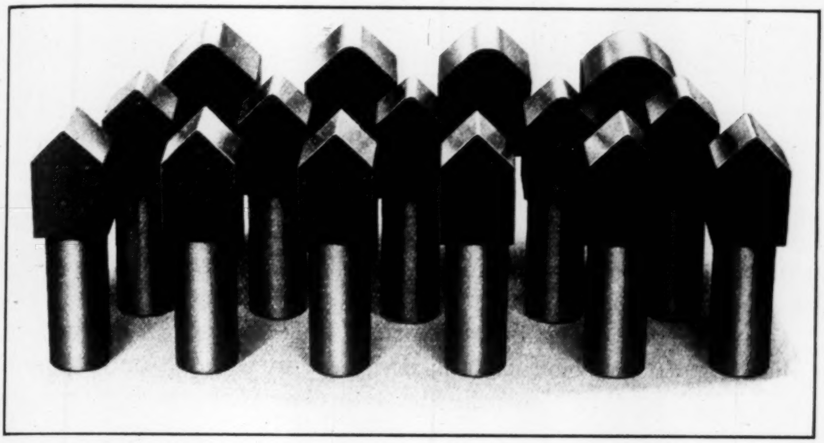


FIG. 2.—Punches.

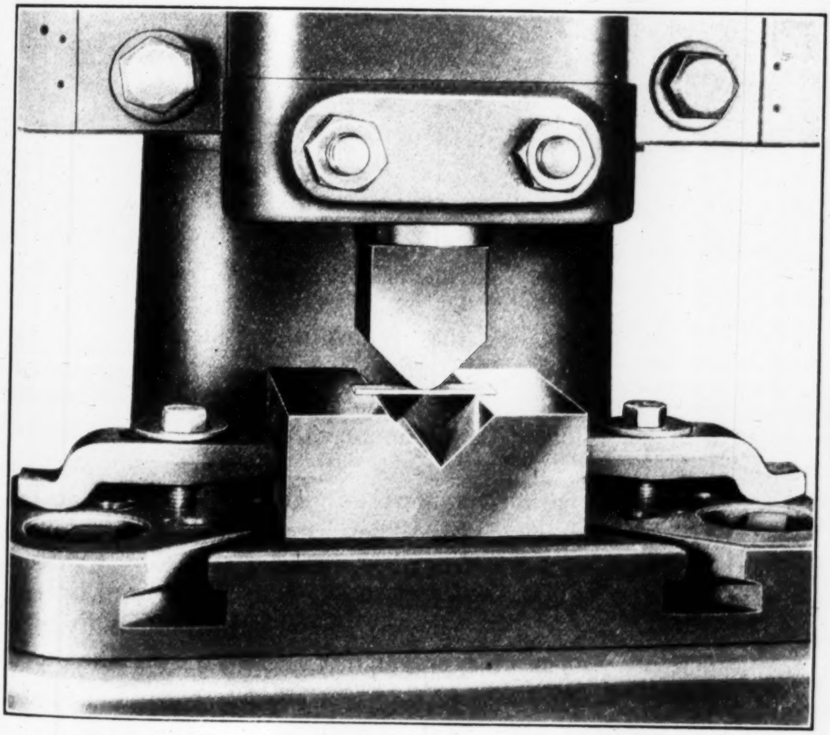
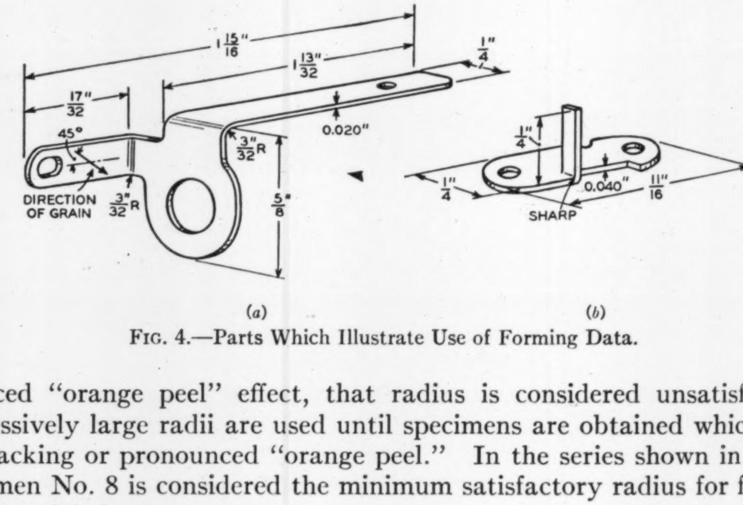


FIG. 3.—Punch and Die Set-Up in Punch Press.

in the present investigation to include additional punches having radii of $\frac{3}{64}$, $\frac{5}{32}$, $\frac{3}{16}$, $\frac{7}{32}$, $\frac{1}{4}$, $\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$ and 1 in. These punches, shown in Fig. 2, were all accurately ground and checked with special contour gages to insure the accuracy of the various radii.

In use, the distance between the punch and the die at the end of the downward stroke is adjusted to the approximate thickness of the material under investigation. The specimen is then placed unclamped on a die having a sharp 90-deg. V groove as shown in Fig. 3 and the specimen formed in the usual manner on a punch press. The press used had a capacity of 25 tons near the bottom of the stroke, a stroke of $1\frac{1}{2}$ in. and a speed of 120 r.p.m.

A series of five specimens are formed over a punch of a given radius and these formed specimens examined visually by comparison with a standard series such as that shown in Fig. 1 to determine whether or not that radius is satisfactory. If these specimens are cracked or have a pronounced "orange peel" effect, that radius is considered unsatisfactory. Successively large radii are used until specimens are obtained which show no cracking or pronounced "orange peel." In the series shown in Fig. 1, specimen No. 8 is considered the minimum satisfactory radius for forming the material shown.



(a) (b)

FIG. 4.—Parts Which Illustrate Use of Forming Data.

The data on the various non-ferrous metals listed in Table I were obtained by the method described. The recommended minimum forming radii for these materials are tabulated in Tables II to IX. In presenting forming data of this type it must be recognized as pointed out in the previous paper,[2] that many other factors influence the formability of sheet metal. Experience has shown however that these data may be used as a basis for design and manufacture.

A particular example of the use of these forming data in design is shown in Fig. 4 (*a*) and (*b*). In the example shown in Fig. 4 (*a*) the contact spring is made from extra hard temper alloy B nickel silver sheet. It is

TABLE I.—CHEMICAL COMPOSITION LIMITS FOR NON-FERROUS SHEET METALS TESTED.

Alloy	Copper, per cent	Lead, per cent	Iron, per cent	Zinc, per cent	Tin, per cent	Nickel, per cent	Aluminum, per cent	Other elements, per cent	Other Impurities, per cent
High brass	64.50 to 67.50	0.30 max.	0.05 max.	Remainder	—	—	—	—	0.10 max.
Red brass	83.00 to 86.00	0.15 max.	0.05 max.	Remainder	—	—	—	—	0.10 max.
Alloy G brass	70.50 to 73.50	0.10 max.	0.05 max.	Remainder	—	—	—	—	0.10 max.
Clock brass	61.00 to 64.00	1.25 to 2.00	0.06 max.	Remainder	—	—	—	—	0.10 max.
Alloy D brass	73.00 to 76.00	0.25 max.	0.05 max.	Remainder	—	—	—	—	0.10 max.
Alloy E brass	64.00 to 67.00	0.80 to 1.10	0.08 max.	Remainder	—	—	—	—	0.10 max.
Low brass[a]	80.00	—	—	20.00	—	—	—	—	—
A phosphor bronze	94.40 min.	0.05 max.	0.10 max.	0.30 max.	3.80 to 4.80	—	—	0.05 to 0.35 phosphorus, 0.01 max. antimony	Trace
C phosphor bronze	91.0 min.	0.02 max.	0.10 max.	0.20 max.	7.50 to 8.50	0.15 max.	—	0.05 to 0.25 phosphorus, 0.01 max. antimony	Trace
D phosphor bronze	90.0 min.	2.50 to 4.00	0.10 max.	0.20 max.	4.50 to 5.50	—	—	0.05 to 0.25 phosphorus	Trace
A nickel silver	70.50 to 73.50	—	0.35 max.	8.50 to 11.50	—	16.50 to 19.50	—	—	—
B nickel silver	53.50 to 56.50	0.10 max.	0.35 max.	25.50 to 28.50	—	16.50 to 19.50	—	0.50 max. manganese	—
Manganese brass	63.00 to 66.00	0.10 max.	0.10 max.	Remainder	—	—	—	0.75 to 1.25 manganese	0.10 max.
Everdur[a]	96.0	—	—	—	—	—	—	3.0 silicon, 1.0 manganese	—
Duronze[a]	97.0	—	—	—	2.0	—	—	1.0 silicon	—
Herculoy	95.25	—	—	1.0	0.5	—	—	3.25 silicon	—
Copper	99.90 min.	—	—	—	—	—	—	—	0.10 max.
Beryllium-copper[a]	Remainder	—	—	—	—	—	—	2.25 beryllium	—
Nickel-beryllium-copper[a]	Remainder	—	—	—	—	0.5	—	2.50 beryllium	—
Monel metal	26.0 to 30.0	—	3.0 max.	—	—	65.0 to 70.0	—	0.25 max. carbon, 0.25 max. phosphorus 0.25 max. silicon, 1.5 max. manganese	
A nickel	0.25 max.	—	0.5 max.	—	—	99.0 min. (plus cobalt)	—	0.15 max. carbon, 0.10 max. phosphorus 0.35 max. manganese, 0.02 max. sulfur	
Inconel[a]	—	—	6.0	—	—	80.0	—	14.0 max. chromium	
2S Aluminum	0.20 max.	—	1.00 max. (plus silicon)		—	—	Remainder	0.05 max. manganese	0.03 max. including zinc
Aluminum Alloy 3S[b]	0.20 max.	—	0.75 max.				Remainder	1.00 to 1.50 manganese, 0.50 max. silicon	0.03 max. including zinc
Aluminum Alloy 17S[b]	3.15 to 4.50	—	0.50 max.	—			Remainder	0.40 to 1.00 magnesium, 0.40 to 1.00 manganese 0.20 to 0.50 silicon	0.03 max. including zinc
Aluminum Alloy 51S[b]	0.10 max.	—	—	—			Remainder	0.80 to 1.20 silicon, 0.40 to 0.80 magnesium, 0.10 max. manganese	0.03 max. including zinc
Aluminum Alloy 52S[b]	0.10 max.	—	0.45 max. (plus silicon)		—	—	Remainder	2.20 to 2.80 magnesium, 0.15 to 0.35 chromium 0.10 max. manganese	
Aluminum Alloy 53S[b]	0.10 max.	—	—				Remainder	1.00 to 1.50 magnesium, 0.50 to 0.90 silicon, 0.15 to 0.35 chromium, 0.10 max. manganese	0.03 max. including zinc

[a] Nominal composition.
[b] Proposed composition limits

necessary to specify a grain direction of 45 deg. to the longer axis of this part because there are two lugs formed at right angles to each other and because minimum forming radii are necessary from a design standpoint. If this grain direction is not specified, and the contact spring is blanked with the grain direction parallel to the longer axis, it becomes necessary to make this contact spring from half-hard temper alloy B nickel-silver sheet. This is obviously unsatisfactory for a contact spring. Another example is shown by the terminal shown in Fig. 4 (*b*), where the hardest temper of high-brass sheet which could be used for the manufacture of this part is hard temper. If a harder temper of this alloy is to be used, it becomes necessary to use a radius of $\frac{1}{8}$ in. with the range of grain no greater than ±45 deg. to the longer axis of the part.

Conclusions

Data on forming tests are tabulated showing the minimum radii for forming 90-deg. bends in various thicknesses, tempers and alloys of brass, phosphor bronze, nickel silver, aluminum and miscellaneous sheet metals.

Application of the data presented herewith has demonstrated the practicability of using them in the design of formed parts and forming tools.

Acknowledgments.—Messrs. J. P. Guerard and A. H. Falk assisted in the laboratory work. Acknowledgment is also made of the helpful counsel of Messrs. J. R. Townsend and C. H. Greenall and their aid in making this investigation possible

TABLE II.—RECOMMENDED RADII FOR FORMING 90-DEG. BENDS IN HIGH SHEET BRASS.

Tensile strength limits are given in parentheses. Rockwell hardness values are limiting values corresponding to tensile strength limits.

Temper, B. & S. Numbers Hard	Nominal Thickness		Rockwell Hardness "B" Scale	Minimum Suitable Radius of Punch, in.		
	B. & S. Gage	Inches		Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Half hard 2 (53,500 to 63,500 lb. per sq. in.)	Nos. 36 to 11 incl.	0.0050 to 0.0907 incl.	49 to 73[a]	Sharp	Sharp	Sharp
Three-quarter hard 3 (61,000 to 71,000 lb. per sq. in.)	Nos. 36 to 17 incl.	0.0050 to 0.0453 incl.	67 to 80[a]	Sharp	Sharp	Sharp
	No. 16	0.0508	70 to 80	Sharp	Sharp	1/64
	No. 15	0.0571	70 to 80	Sharp	Sharp	1/64
	No. 14	0.0641	70 to 80	1/64	1/64	1/32
	No. 12	0.0808	70 to 80	1/64[b]	1/32[b]	3/32[b]
	No. 11	0.0907	70 to 80	1/64	1/32	3/32
Hard 4 (68,000 to 78,000 lb. per sq. in.)	Nos. 36 to 22 incl.	0.0050 to 0.0253 incl.	75 to 83[a]	Sharp	Sharp	Sharp
	No. 20	0.0320	75 to 83	Sharp	Sharp	1/64
	No. 18	0.0403	78 to 85	Sharp	Sharp	1/32
	No. 17	0.0453	78 to 85	Sharp	1/64	1/16
	No. 16	0.0508	78 to 85	Sharp	1/64	3/32
	No. 15	0.0571	78 to 85	1/64	1/32	3/32
	No. 14	0.0641	78 to 85	1/32	1/32	3/32
	No. 12	0.0808	78 to 85	1/32	1/16	3/32
	No. 11	0.0907	78 to 85	3/32	1/8	1/4
	No. 10	0.1019	78 to 85	1/4	1/4	1/4
	No. 9	0.1144	78 to 85	1/4	1/4	1/4
Extra hard 6 (79,000 to 88,500 lb. per sq. in.)	No. 24	0.0201	83 to 87	1/64	1/32	3/32
	No. 20	0.0320	83 to 87	1/32	1/16	3/16
	No. 19	0.0359	83 to 87	3/64[b]	3/32	3/16
	No. 18	0.0403	85 to 89	3/64	1/8	3/16
	No. 17	0.0453	85 to 89	1/16	1/8[b]	3/16[b]
	No. 16	0.0508	85 to 89	1/16	1/8	3/16
	No. 15	0.0571	85 to 89	3/32	5/32	1/4
	No. 14	0.0641	85 to 89	1/8	5/32	3/8
	No. 13	0.0720	85 to 89	1/8	1/4	3/8
	No. 11	0.0907	85 to 89	1/4	3/8	1/2
Spring 8 (86,000 to 95,000 lb. per sq. in.)	No. 31	0.0089		Sharp	1/64	3/32
	No. 30	0.0100		Sharp	1/64	3/32
	No. 26	0.0159		1/64	1/16	5/32
	No. 24	0.0201	85 to 89	1/32	3/32	7/32
	No. 22	0.0253	85 to 89	1/32	5/32	>1/4
	No. 20	0.0320	85 to 89	1/32	5/32	>1/4
	No. 18	0.0403	88 to 92	3/64	7/32	>1/4
	No. 16	0.0508	88 to 92	3/32	7/32	>1/4
	No. 14	0.0641	88 to 92	1/8	1/4	>1/4
Extra spring 10 (89,500 to 98,500 lb. per sq. in.)	No. 22	0.0253	86 to 90	3/64	5/32	>1/4

[a] Rockwell hardness limits do not apply to material less than 0.0201 in. thick.
[b] Interpolated from forming data on other gages.

TABLE III.—RECOMMENDED RADII FOR FORMING 90-DEG. BENDS IN RED BRASS SHEET.

Proposed tensile strength limits are given in parentheses. Rockwell hardness values are limiting values corresponding to proposed tensile strength limits.

These values have been determined independently by the Western Electric Co., but data have not been published.

Temper, B. & S. Numbers Hard	Nominal Thickness		Rockwell Hardness "B" Scale	Minimum Suitable Radius of Punch, in.		
	B. & S. Gage	Inches		Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Drawing anneal 0 (33,500 to 43,500 lb. per sq. in.)	Nos. 36 to 14 incl.	0.0050 to 0.0641 incl.		Sharp	Sharp	Sharp
Half hard 2 (46,500 to 56,500 lb. per sq. in.)	Nos. 36 to 26 incl.	0.0050 to 0.0159 incl.		Sharp	Sharp	Sharp
	Nos. 24 to 16 incl.	0.0201 to 0.0508 incl.	43 to 71	Sharp	Sharp	1/64
	No. 14	0.0641	48 to 71	1/32	1/32	1/32
Hard 4 (60,500 to 70,500 lb. per sq. in.)	Nos. 36 to 28 incl.	0.0050 to 0.0126		Sharp	Sharp	Sharp
	No. 26	0.0159		Sharp	1/64	1/64
	No. 24	0.0201	66 to 78	Sharp	1/64	1/64
	No. 22	0.0253	66 to 78	Sharp	1/64	1/32
	No. 20	0.0320	66 to 78	Sharp	1/64	1/16
	No. 18	0.0403	73 to 81	1/64	1/32	3/32
	No. 16	0.0508	73 to 81	1/64	1/32	3/32
	No. 14	0.0641	73 to 81	1/32	1/16	1/8
Extra hard 6 (70,000 to 79,500 lb. per sq. in.)	No. 36	0.0050		Sharp	Sharp	1/64
	No. 34	0.0063		Sharp	Sharp	1/64
	No. 32	0.0080		Sharp	Sharp	1/32
	No. 30	0.0100		Sharp	Sharp	1/32
	No. 28	0.0126		Sharp	1/64	1/16
	No. 26	0.0159		Sharp	1/32	3/32
	No. 24	0.0201	74 to 84	1/64	1/32	1/8
	No. 22	0.0253	74 to 84	1/64	1/16	5/32
	No. 20	0.0320	74 to 84	1/64	1/16	3/16
	No. 18	0.0403	79 to 85	1/16	3/32	7/32
	No. 16	0.0508	79 to 85	1/16	1/8	3/8
	No. 14	0.0641	79 to 85	1/16	3/16	3/8
Spring 8 (75,000 to 84,000 lb. per sq. in.)	No. 36	0.0050		Sharp	1/64	1/64
	No. 34	0.0063		Sharp	1/64	1/32
	No. 32	0.0080		Sharp	1/64	1/16
	No. 30	0.0100		Sharp	1/64	3/32
	No. 28	0.0126		Sharp	1/32	1/8
	No. 26	0.0159		1/64	1/32	3/16
	No. 24	0.0201	79 to 88	1/64	3/32	7/32
	No. 22	0.0253	79 to 88	1/32	1/8	3/8
	No. 20	0.0320	79 to 88	1/16	1/8	3/8
	No. 18	0.0403	82 to 88	1/16	3/16	1/2
	No. 16	0.0508	82 to 88	1/16	1/4	1/2
	No. 14	0.0641	82 to 88	3/32	3/8	1/2

TABLE IV.—RECOMMENDED RADII FOR FORMING 90-DEG. BENDS IN ALLOY G BRASS.

N.D = Not determined.
Tensile strength limits are given in parentheses.
Rockwell hardness values are limiting values corresponding to tensile strength limits.

Temper, B. & S. Numbers Hard	Nominal Thickness		Rockwell Hardness "B" Scale	Minimum Suitable Radius of Punch, in.		
	B. & S. Gage	Inches		Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Half hard 2 (56,500 to 66,500 lb. per sq. in.)	Nos. 36 to 26 incl.	0.0050 to 0.0159 incl.		Sharp	Sharp	Sharp
	Nos. 24 to 20 incl.	0.0201 to 0.0320 incl.	57 to 73	Sharp	Sharp	Sharp
	No. 18	0.0403	63 to 76	Sharp	Sharp	Sharp
	No. 16	0.0508	63 to 76	Sharp	Sharp	Sharp
	No. 14	0.0641	63 to 76	1/64	N.D.	N.D.
Hard 4 (71,000 to 81,000 lb. per sq. in.)	Nos. 36 to 26 incl.	0.0050 to 0.0159 incl.		Sharp	Sharp	Sharp
	Nos. 24 to 20 incl.	0.0201 to 0.0320 incl.	77 to 84	1/64	1/32	3/64
	No. 18	0.0403	80 to 86	1/64	1/32[a]	3/64
	No. 16	0.0508	80 to 86	1/32[a]	1/32[a]	1/8
	No. 14	0.0641	80 to 86	1/32	1/32	1/8
Extra hard 6 (82,500 to 91,500 lb. per sq. in.)	No. 36	0.0050		Sharp	Sharp	Sharp
	No. 34	0.0063		Sharp	Sharp	Sharp
	No. 32	0.0080		Sharp	Sharp	1/64
	No. 30	0.0100		Sharp	Sharp	1/32
	No. 28	0.0126		Sharp	Sharp	1/32
	No. 26	0.0159		Sharp	1/64	1/16
	No. 24	0.0201	85 to 89	1/64	3/64	1/8
	No. 22	0.0253	85 to 89	1/64	1/16	5/32
	No. 20	0.0320	85 to 89	1/32	3/32	5/32
	No. 18	0.0403	87 to 91	1/32	5/32	7/32
	No. 16	0.0508	87 to 91	3/64	3/16	>1/4
	No. 14	0.0641	87 to 91	1/16	>1/4	>1/4
Spring 8 (90,500 to 99,500 lb. per sq. in.)	No. 36	0.0050		Sharp	Sharp	1/64[a]
	No. 34	0.0063		Sharp	Sharp	1/64
	No. 32	0.0080		Sharp	1/64	1/32
	No. 30	0.0100		Sharp	1/64	3/32
	No. 28	0.0126		Sharp	1/16	3/32
	No. 26	0.0159		Sharp	1/16[a]	1/8
	No. 24	0.0201	88 to 92	1/32	1/8	7/32
	No. 22	0.0253	88 to 92	1/32	1/8	7/32
	No. 20	0.0320	88 to 92	3/64	N.D.	N.D.
	No. 18	0.0403	90 to 94	1/16	>1/4	>1/4
	No. 16	0.0508	90 to 94	1/8	>1/4	>1/4
	No. 14	0.0641	90 to 94	1/8	>1/4	>1/4
Extra spring 10 (95,000 to 104,000 lb. per sq. in.)	No. 36	0.0050		Sharp	Sharp	1/32
	No. 34	0.0063		Sharp	Sharp	1/32
	No. 32	0.0080		Sharp[a]	3/64	1/16
	No. 30	0.0100		Sharp	1/16	3/32
	No. 28	0.0126		Sharp	1/16	N.D.
	No. 26	0.0159		Sharp	1/16	7/32
	No. 22	0.0253	90 to 94	3/64	5/32	>1/4
	No. 20	0.0320	90 to 94	3/64	3/16	>1/4
	Nos. 18 to 14 incl.	0.0403 to 0.0641 incl.	92 to 96	1/8	>1/4	>1/4

[a] Interpolated from forming data on other gages.

TABLE V.—RECOMMENDED RADII FOR FORMING 90-DEG. BENDS IN ALLOY C PHOSPHOR BRONZE SHEET.

N.D. = Not determined.
Tensile strength limits are given in parentheses.
Rockwell hardness values are limiting values corresponding to tensile strength limits.

Temper, B. & S. Numbers Hard	Nominal Thickness		Rockwell Hardness "G" Scale	Minimum Suitable Radius of Punch, in.		
	B. & S. Gage	Inches		Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Half hard 2 (69,000 to 84,000 lb. per sq. in.)	Nos. 36 to 14 incl.	0.0050 to 0.0641 incl.	[b]	Sharp	Sharp	Sharp
	No. 13	0.0720	47 to 68	Sharp	Sharp	N.D.
		0.1250	47 to 68	1/8	1/8	N.D.
Hard 4 (85,000 to 100,000 lb. per sq. in.)	No. 36	0.0050		Sharp	1/64	1/64
	No. 34	0.0063		Sharp	N.D.	1/64
	No. 32	0.0080		Sharp	N.D.	1/64
	No. 30	0.0100		Sharp	N.D.	1/32
	No. 28	0.0126		Sharp	N.D.	1/32
	No. 26	0.0159		Sharp	N.D.	N.D.
	No. 24	0.0201	65 to 74	Sharp	N.D.	1/16
	No. 22	0.0253	65 to 74	Sharp	N.D.	N.D.
	No. 18	0.0403	69 to 77	1/32	1/8[a]	N.D.
	No. 16	0.0508	69 to 77	3/32	1/8	3/16
	No. 14	0.0641	69 to 77	N.D.	1/8	1/4
Extra hard 6 (97,000 to 111,500 lb. per sq. in.)	No 36	0.0050		Sharp	N.D.	1/32
	No. 34	0.0063		Sharp	N.D.	1/32
	No. 32	0.0080		Sharp	N.D.	3/64
	No. 30	0.0100		Sharp	N.D.	N.D.
	No. 28	0.0126		Sharp	N.D.	N.D.
	No. 26	0.0159		Sharp	N.D	N.D.
	No. 24	0.0201	73 to 79	Sharp	N.D.	1/4
	No. 22	0.0253	73 to 79	1/32	N.D.	N.D.
	No. 20	0.0320	73 to 79	1/32	N.D.	N.D.
	No. 18	0.0403	76 to 82	3/64	5/32	>1/4
	No. 16	0.0508	76 to 82	N.D.	3/16	>1/4
	No. 14	0.0641	76 to 82	5/32	>1/4	>1/4
Spring 8 (105,000 to 118,500 lb. per sq. in.)	No. 36	0.0050		Sharp	1/64	3/64
	No. 34	0.0063		Sharp	1/32	3/32
	No. 32	0.0080		Sharp	3/64[a]	3/32
	No. 30	0.0100		1/64	1/16	1/8
	No. 28	0.0126		1/64	3/32	5/32
	No. 26	0.0159		1/32	3/32	5/32
	No. 25	0.0179		1/32	1/8	7/32
	No. 24	0.0201	76 to 82	1/32	1/8	1/4
	No. 22	0.0253	76 to 82	1/32	5/32	3/8
	No. 20	0.0320	76 to 82	3/32	3/16	1/2
	No. 19	0.0359	76 to 82	3/32[a]	7/32	1/2
	No. 18	0.0403	79 to 85	3/32	1/4	1/2
	No. 17	0.0453	79 to 85	1/8	3/8	1/2
	No. 16	0.0508	79 to 85	N.D.	3/8	1/2
	No. 14	0.0641	79 to 85	5/32	3/8	3/4
	No. 11	0.0907	79 to 85	7/32	1/2	3/4
Extra spring 10 (109,500 to 122,000 lb. per sq. in.)	No. 36	0.0050		Sharp	N.D.	1/16
	No. 32	0.0080		1/64	N.D.	3/32
	No. 28	0.0126		1/64	N.D.	N.D.
	No. 26	0.0159		1/64	N.D.	N D.
	No. 24	0.0201	78 to 83	1/32	N.D.	N.D.
	No. 14	0.0641	81 to 86	5/32	>1/4	>1/4

[a] Interpolated from forming data on other gages.
[b] Rockwell hardness 20 to 24, B. & S. gage 40 to 64; heavier than 20, B. & S. gage 47 to 68.

TABLE VI.—RECOMMENDED RADII FOR FORMING 90-DEG. BENDS IN ALLOY A NICKEL SILVER SHEET.

N.D. = Not determined. Tensile strength limits are given in parentheses.

Temper, B. & S. Numbers Hard	Nominal Thickness		Minimum Suitable Radius of Punch, in.		
	B. & S. Gage	Inches	Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Half hard 2 (65,500 to 73,500 lb. per sq. in.)	Nos. 36 to 19 incl.	0.0050 to 0.0359 incl.	Sharp	Sharp	Sharp
	No. 18	0.0403	N.D.	1/64	1/32
	No. 16	0.0508	Sharp	1/64	N.D.
	No. 14	0.0641	N.D.	N.D.	3/32
	No. 13	0.0720	3/32	3/32	3/32
	No. 11	0.0907	3/32	3/32	1/8
Hard 4 (75,000 to 82,000 lb. per sq. in.)	Nos. 36 to 26 incl.	0.0050 to 0.0159 incl.	Sharp	Sharp	Sharp
	No. 24	0.0201	Sharp	Sharp	1/64
	No. 22	0.0253	Sharp	Sharp	N.D.
	No. 20	0.0320	1/16	1/16	1/16
	No. 18	0.0403	1/16	1/16	1/16
	No. 16	0.0508	1/16	1/16	3/32
Extra hard 6 (81,000 to 88,000 lb. per sq. in.)	Nos. 36 to 26 incl.	0.0050 to 0.0159 incl.	Sharp	Sharp	N.D.
	No. 22	0.0253	1/64	1/32	N.D.
	No. 20	0.0320	1/8	1/8	1/8
	No. 18	0.0403	1/8	1/8	3/16
	No. 16	0.0508	5/32	5/32	7/32
Spring 8	Nos. 36 to 32 incl.	0.0050 to 0.0080 incl.	Sharp	Sharp	N.D.
	No. 30	0.0100	Sharp	1/64	N.D.
	No 28	0.0126	1/64	1/64[a]	N.D.
	No. 26	0.0159	1/64	1/64	N.D.
	No. 24	0.0201	3/64	3/32	3/16
	No. 22	0.0253	3/64	3/16[a]	N.D.
	No. 20	0.0320	5/32	3/16	N.D.
	No. 18	0.0403	5/32	3/16	7/32
	No. 16	0.0508	5/32	7/32	N.D.
	No. 14	0.0641	>1/4	>1/4[a]	>1/4
Extra spring 10	No. 36	0.0050	Sharp[a]	Sharp	N.D.
	No. 34	0.0063	Sharp	Sharp	N.D.
	No. 32	0.0080	Sharp	1/64	N.D.
	No. 30	0.0100	Sharp	1/64	N.D.
	No. 28	0.0126	1/64	N.D.	N.D.
	No. 26	0.0159	1/64	1/32	N.D.
	No. 22	0.0253	3/64	1/8	N.D.
	No. 20	0.0320	5/32	7/32	N.D.
	No. 18	0.0403	5/32	7/32	1/4
	No. 16	0.0508	5/32	1/4	N.D.

[a] Interpolated from forming data on other gages.

TABLE VII.—RECOMMENDED RADII FOR FORMING 90-DEG. BENDS IN ALLOY B NICKEL SILVER SHEET.

Tensile strength limits are given in parentheses. Rockwell hardness values are limiting values correspondin to tensile strength limits.

Temper, B. & S. Numbers Hard	Nominal Thickness		Rockwell Hardness "G" Scale	Minimum Suitable Radius of Punch, in.		
	B. & S. Gage	Inches		Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Half hard 2 (78,000 to 93,000 lb. per sq. in.)	Nos. 36 to 26 incl.	0.0050 to 0.0159 incl.		Sharp	Sharp	Sharp
	No. 24	0.0201	48 to 67	Sharp	Sharp	Sharp
Hard 4 (92,000 to 106,500 lb. per sq. in.)	No. 24	0.0201	66 to 75	1/32	1/32	3/32
Extra hard 6 (102,000 to 115,000 lb. per sq. in.)	No. 36	0.0050		Sharp	Sharp	Sharp
	No. 34	0.0063		Sharp	Sharp	Sharp
	No. 32	0.0080		1/64	1/64	1/64
	No. 30	0.0100		1/64	1/64	1/64
	No. 28	0.0126	69 to 78	1/64	1/64	1/64
	No. 27	0.0142	69 to 78	1/64	1/64	1/32
	No. 26	0.0159	69 to 78	1/32	3/64	1/8
	No. 25	0.0179	69 to 78	1/32	3/64	1/8
	No. 24	0.0201	72 to 79	3/64	3/32	1/8
	No. 23	0.0226	72 to 79	1/16	3/32	1/8
	No. 22	0.0253	72 to 79	3/32	3/32	1/8
	No. 21	0.0285	72 to 79	3/32	3/32	3/16
	No. 20	0.0320	72 to 79	3/32	3/32	3/16
	No. 19	0.0359	72 to 79	1/8	1/8	3/16
	No. 18	0.0403	75 to 82	1/8	5/32	3/16
	No. 16	0.0508	75 to 82	1/8	5/32	7/32
	No. 14	0.0641	75 to 82	5/32	3/16	3/8
	No. 12	0.0808	75 to 82	1/4	1/4	3/8
	No. 11	0.0907	75 to 82	3/8	3/8	3/8
		0.1250	75 to 82	>1/4	>1/4	>1/4

Table VIII.—Recommended Radii for Forming 90-deg. Bends in Various Sheet Aluminum Alloys.

Proposed tensile strength limits are shown in parentheses.

Alloy	Temper	Nominal Thickness		Minimum Suitaable Radius of Punch, in.		
		B. & S. Gage	Inches	Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
2SH	Hard (22,000 lb. per sq. in. min.)	No. 14	0.0641	3/32	1/8	1/8
		No. 8	0.1285	5/32	5/32	5/32
		No. 5	0.1819	3/16	3/16	3/16
3S 1/2H	Half hard (19,000 to 24,000 lb. per sq. in.)	No. 14	0.0641	Sharp	Sharp	1/64
		No. 8	0.1285	Sharp	Sharp	1/64
		No. 5	0.1819	3/16	3/16	7/32
3SH	Hard (27,000 to 33,000 lb. per sq. in.)	No. 26	0.0159	Sharp	Sharp	Sharp
		No. 20	0.0320	Sharp	Sharp	1/64
		No. 14	0.0641	5/32	5/32	3/8
		No. 8	0.1285	5/32	5/32	3/8
		No. 5	0.1819	3/16	3/16	1/2
17ST	Heat treated (55,000 lb. per sq. in. min.)	No. 28	0.0126	1/64	1/64	1/64
		No. 24	0.0201	1/32	1/32	1/32
		No. 20	0.0320	1/16	1/16	1/16
		No. 14	0.0641	1/8	1/8	1/8
		No. 10	0.1019	5/32	3/16	3/16
		No. 8	0.1285	3/16	3/16	3/8
51SW	Heat treated but not aged (30,000 to 40,000 lb. per sq. in.)	No. 26	0.0159	Sharp	Sharp	1/64
		No. 24	0.0201	1/64	1/64	1/64
		No. 20	0.0320	1/64	1/64	1/64
		No. 14	0.0641	1/32	1/32	1/16
		No. 8	0.1285	3/64	1/16	3/32
51ST	Heat treated (40,000 lb. per sq. in. min.)	No. 28	0.0126	1/32	1/32	3/64
		No. 24	0.0201	1/16	1/16	1/16
		No. 20	0.0320	3/32	3/32	3/32
		No. 14	0.0641	5/32	3/16	3/16
		No. 8	0.1285	3/8	1/2	1/2
52SO	Annealed (31,000 lb. per sq. in. max.)	No. 8	0.1285	Sharp	Sharp	Sharp
		No. 5	0.1819	Sharp	Sharp	Sharp
52S 1/4H	Quarter hard (31,000 to 36,000 lb. per sq. in.)	No. 28	0.0126	Sharp	1/64	1/64
		No. 24	0.0201	1/64	1/64	1/32
		No. 20	0.0320	1/64	1/64	1/32
		No. 14	0.0641	1/64	1/64	1/32
		No. 8	0.1285	1/64	1/32	1/32
		No. 5	0.1819	1/32	1/32	3/64
52S 1/2H	Half hard (34,000 to 39,000 lb. per sq. in.)	No. 28	0.0126	1/64	1/64	1/64
		No. 26	0.0159	1/64	1/64	1/64
		No. 24	0.0201	1/64	1/64	1/32
		No. 20	0.0320	1/64	1/64	1/32
		No. 14	0.0641	1/32	1/32	3/64
		No. 8	0.1285	1/8	1/8	1/8
52S 3/4H	Three quarter hard (37,000 to 42,000 lb. per sq. in.)	No. 28	0.0126	1/64	1/64	1/32
		No. 26	0.0159	1/64	1/64	1/32
		No. 24	0.0201	1/64	1/32	1/32
		No. 20	0.0320	1/64	1/32	1/32
		No. 14	0.0641	3/32	1/8	1/8
		No. 8	0.1285	7/32	7/32	7/32
52SH	Hard (39,000 to 44,000 lb. per sq. in.)	No. 28	0.0126	1/64	1/64	1/32
		No. 26	0.0159	1/64	1/32	1/32
		No. 24	0.0201	1/64	1/32	1/32
		No. 20	0.0320	1/32	1/32	3/64
		No. 14	0.0641	5/32	5/32	3/16
		No. 8	0.1285	5/32	3/8	3/8
53SW	Heat treated but not aged (28,000 to 38,000 lb. per sq. in.)	No. 26	0.0159	1/64	1/64	1/64
		No. 24	0.0201	1/64	1/64	1/64
		No. 20	0.0320	1/64	1/64	1/64
		No. 14	0.0641	1/64	1/64	1/32
		No. 8	0.1285	1/64	1/64	3/64
53ST	Heat treated (35,000 lb. per sq. in. min.)	No. 28	0.0126	1/64	1/32	1/32
		No. 24	0.0201	3/64	3/64	3/64
		No. 20	0.0320	3/64	3/64	3/64
		No. 14	0.0641	3/64	3/64	1/16
		No. 8	0.1285	3/32	1/8	5/32

Table IX.—Indicated Radii for Forming 90-deg. Bends in Miscellaneous Sheet Metals.

Tensile strength limits are shown in parentheses. Rockwell hardness values correspond to tensile strength limits.

Alloy	Temper, B. & S. Numbers Hard	Nominal Thickness		Rockwell Hardness, "G" Scale	Minimum Suitable Radius of Punch, in.		
		B. & S. Gage	Inches		Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling
Clock brass	Hard 4 (68,000 to 78,000 lb. per sq. in.)	No. 24	0.0201	75 to 83[b]	Sharp	1/32	1/32
	Spring 8 (85,000 to 94,500 lb. per sq. in.)	No. 24	0.0201	85 to 89[b]	1/16	5/32	>1/4
D brass	Hard 4	No. 24	0.0201		1/32	3/64	7/32
	Spring 8	No. 24	0.0201		1/32	3/32	7/32
E brass	Hard 4 (68,000 to 78,000 lb. per sq. in.)	No. 24	0.0201	75 to 83[b]	3/64	3/64	1/16
	Spring 8	No. 24	0.0201		1/16	5/32	1/4
Low brass	Hard 4	No. 24	0.0201		1/32	3/64	1/16
	Spring 8	No. 24	0.0201		1/32	1/8	3/16
A Phosphor bronze	Half hard 2 (55,000 to 70,000 lb. per sq. in.)	No. 24	0.0201	15 to 51	Sharp	Sharp	Sharp
	Hard 4 (72,000 to 87,000 lb. per sq. in.)	No. 24	0.0201	53 to 64	Sharp	1/32	1/16
	Spring 8 (91,000 to 105,000 lb. per sq. in.)	No. 24	0.0201	67 to 73	1/64	3/32	3/16
D Phosphor bronze	Hard 4 (71,000 to 85,000 lb. per sq. in.)	No. 24	0.0201		1/32	3/32	7/32
	Spring 8 (91,000 to 101,000 lb. per sq. in.)	No. 24	0.0201		1/16	7/32	>1/4
Manganese brass	Hard 4	No. 24	0.0201		Sharp	1/64	3/64
	Extra hard 6	No. 24	0.0201		1/32	1/16	1/8
	Spring 8	No. 24	0.0201		1/32	1/8	1/4
Copper silicon alloys[a]	Hard 4	No. 24	0.0201		1/32	1/32	1/16
	Spring 8	No. 24	0 0201		3/64	3/32	3/16
Copper	Half hard 2 (37,000 to 44,000 lb. per sq. in.)	No. 24	0.0201	30 to 50[b]	1/32	1/32	3/64
	Extra hard 6 (46,000 to 55,000 lb. per sq. in.)	No. 24	0.0201	50 to 62[b]	1/64	1/32	1/32
Monel metal	Hard 5	No. 24	0.0201		1/32	3/64	1/8
	Full hard 7	No. 24	0.0201		1/32	3/64	5/32
Grade A nickel	Hard 4	No. 24	0.0201		1/32	1/32	3/16
	Spring 8	No. 24	0.0201		1/32	3/32	3/16
Inconel	Soft 0	No. 24	0.0201		Sharp	Sharp	Sharp
	Hard 4	No. 24	0.0201		Sharp	Sharp	Sharp
Heated 1 hr. at 800 C., Quenched in Water							
Beryllium-copper	Soft 0	No. 18	0.0403		Sharp	Sharp	Sharp
	Half hard 2	No. 18	0.0403		1/16	3/32	1/8
	Hard 4	No. 18	0.0403		3/16	7/32	>1/4
Nickel-beryllium copper	Soft 0	No. 26	0.0159		1/64	1/64	1/64
	Quarter hard 1	No. 26	0.0159		1/32	1/32	1/32
		No. 18	0.0403		3/32	1/16	1/8
	Half hard 2	No. 26	0.0159		1/32	1/32	1/32
	Quarter hard 1 (aged after rolling)	No. 18	0.0403		1/4	>1/4	>1/4

[a] Includes Everdur, Duronze and Herculoy.

[b] "B" Scale.

DISCUSSION

Mr. W. F. Burchfield.[1]—We have conducted similar tests on monel metal, nickel and Inconel cold-rolled strip using the same set of forming tools used for the investigation made by the Western Electric Co.[2] Our tests conducted on strip 0.031, 0.062 and 0.125 in. thick, heavier than the material used by Mr. Gohn, indicated that sharp bends in all directions could be made in tempers half-hard and softer in monel metal, nickel and Inconel.

Mr. Gohn shows hardness tempers of our metals as having been obtained by cold rolling so many B. & S. numbers hard. Although this was the practice used in rolling strips for Mr. Gohn's investigation, it is not our standard mill practice. We roll to U.S. gages and obtain our tempers by cold rolling sufficiently to meet our hardness specifications.

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[1] Technical Service, Development and Research Division, The International Nickel Co., New York City.

[2] W. A. Straw, M. D. Helfrick and C. R. Fischrupp, "Forming Properties of Thin Sheets of Some Non-Ferrous Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Division, p. 317 (1931).

STRESS-RELIEF ANNEALING HIGH-STRENGTH MONEL METAL PLATE[1]

By Peter R. Kosting[2]

Synopsis

The effect of temperature of annealing and time at temperature upon the tensile properties of high strength monel metal plate, ½ in. thick, is discussed. Plates from 4 heats were studied. All plates were hot rolled but finished at a low temperature except some plates from one heat which were cold rolled 10 per cent. Extensometers of 0.0002 and 0.00002 in. sensitivity were used for determining yield strength, 0.00 per cent set. All plates showed maximum increase in yield strength, 0.00 per cent set, in the temperature range 550 to 600 C. (1020 to 1110 F.). Yield strengths at higher per cent sets reached maximum values at lower temperatures: the greater the effect of cold work the lower was the annealing temperature at which maximum values were obtained. The value of the yield strength, 0.00 per cent set, is appreciably affected by internal strains. It is apparent that this monel metal plate must be heated to 550 to 600 C. (1020 to 1110 F.) to be stress-relieved.

Introduction:

In developing the technique of welding thick monel metal plate, a suitable stress-relief annealing temperature had to be determined. The plate involved was of high strength, meeting the following specification limits: tensile strength 90,000 lb. per sq. in., minimum; yield strength, 0.05 per cent set, 55,000 lb. per sq. in., minimum; elongation 18 per cent, minimum; and reduction of area 30 per cent, minimum. In general the plate is hot rolled but finished at a lower temperature than usual. It is possible to obtain these physical properties by cold rolling. The size of the plate and the power of the rolling mills are factors that govern the selection of fabrication method to be used.

There are several methods suggested[3] in the literature that might be suitable for determining a stress-relief annealing temperature. Heyn[4] discussed how internal stresses and their distribution affected the yield strength, 0.00 per cent set (a more descriptive term for proportional limit). Moore and Beckinsale[5] and others have reported that annealing metal at intermediate temperatures increased to a marked degree the yield strength.

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[1] Publication approved by Chief of Ordnance, U. S. Army.

[2] Chemical Engineer, Watertown Arsenal, Watertown, Mass.

[3] C. H. Barrett, "Internal Stresses," *Metals and Alloys*, Vol. 5, pp. 131, 154, 170, 196, 224 (1934).

[4] E. Heyn, "Internal Strains in Cold Wrought Metals, and Some Troubles Caused Thereby," *Journal*, Inst. Metals, Vol. 12, p. 3 (1914).

[5] H. Moore and S. Beckinsale, "The Prevention of Season Cracking in Brass by the Removal of Internal Strains," *Transactions*, Faraday Soc., Vol. 17, p. 162 (1921).

0.00 per cent set. It is general experience at Watertown Arsenal that any metal which showed instability of dimensions upon machining did not have as high a yield strength, 0.00 per cent set, as it would have had, had it been properly stress-relief annealed.

The work of Pilling,[6] Crawford and Worthington[7] and Geiger[8] indicated that 275 to 300 C. (525 to 570 F.) is used for stress relief annealing cold-worked monel metal. Arnott[9] reported that annealing at 700 C. (1290 F.) did not appreciably change the properties of rolled bar stock. His data showed that between 500 and 600 C. (930 and 1110 F.) an improvement in properties occurred, that is, yield strength and tensile strength increased with negligible change in elongation and reduction of area.

It was believed that if the effect of temperature of annealing and time at temperature upon the tensile properties of the high strength plate were studied the greatest amount of information of a general nature would be obtained, especially if particular attention were paid to the yield strength, 0.00 per cent set. This paper gives the results of such a study.

TABLE I.—ANALYSIS OF $\frac{1}{2}$-IN. MONEL METAL PLATE.

Watertown Arsenal Designation	Plate	Carbon, per cent	Sulfur, per cent	Silicon, per cent	Manganese, per cent	Iron, per cent	Copper, per cent	Nickel, per cent
Mn........	....	0.18	....	0.08	1.09	2.08	29.1	67.29
No. 2.......	No. 1344-2[a]......	0.11	0.007	0.06	0.86	1.33	32.13	65.48
No. 1.......	No. 1521-2[a].. ...	0.10	0.009	0.07	0.89	1.33	30.68	66.90
No. 3 No. 6R....	No. 1316-2[a]. No. 1316-R[a]....	0.15	0.007	0.07	1.07	1.03	31.39	66.26

[a] By manufacturer.

Material:

The analyses of four heats of plate used in this study are given in Table I.

Test Procedure:

Strips 4 to 5 in. by $\frac{1}{2}$ by $\frac{1}{2}$ in. were annealed at the indicated temperature for the indicated time in an electric furnace. They were placed in the hot furnace and 10 min. were allowed for them to come up to temperature. All were furnace cooled.

From these strips 0.357-in. diameter tension test specimens were machined. The physical properties were determined on a hydraulic

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[6] N. B. Pilling, "Effect of Cold Working on the Izod Notched-Bar Impact Value of Monel Metal," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 576 (1932).

[7] C. A. Crawford and Robert Worthington, "Nickel and Nickel Alloys Other than the Nickel-Chromium-Iron Group," Symposium on Effect of Temperature on the Properties of Metals, published jointly by the American Society for Testing Materials and The American Society of Mecnanical Engineers, p. 557 (1931). (Available as separate publication.)

[8] G. F. Geiger, "Working Ranges of Nickel Copper Alloys," *Inco News*, Vol. 12, No. 4, p. 16 (1935).

[9] J. Arnott, "Monel Metal," *Journal*, Inst. Metals, Vol. 23, No. 1, p. 545 (1920).

Amsler machine of 50,000 lb. capacity. Stress-strain diagrams, of which Fig. 1 is typical, were obtained for all plates except plate Mn on a Baldwin-Southwark Recorder. The sensitivity of its extensometer was 0.00002 in. Data on plate Mn were obtained with an extensometer the sensitivity of which was 0.0002 in.

Test Results:

Typical data are given in Table II and in Figs. 2, 3 and 4.

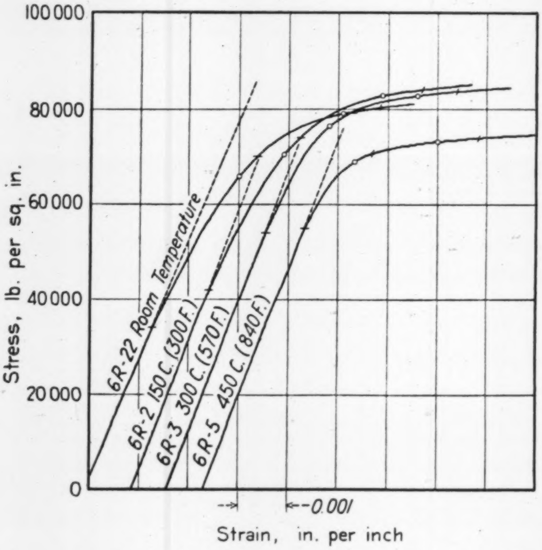

FIG. 1.—Stress-Strain Diagram for ½-in. Monel Metal Plate No. 1316-R.
Stress relief anneal, cold-rolled plates, 1 hr. at temperature, 0.357-in. longitudinal bars.

Discussion:

Study of the stress-strain curves reveals that the deviation from a straight line is small and that the rate at which the curves deviate is slow for metal containing internal stresses. The yield strength at 0.00 per cent set for such metal will depend upon the sensitivity of the extensometer, being appreciably lower the greater the sensitivity. As the per cent set is increased, the effect of sensitivity of extensometer becomes smaller.

Table III shows the difference in physical properties of unstress-relieved monel metal due to using extensometers whose sensitivities are 0.0002 and 0.00002 in.

As the internal stresses are relieved, the departure of the stress-strain curves from straight lines becomes sharper and sharper so that the effect of sensitivity of extensometer should become smaller. As the sensitivity of the extensometer increases, however, difficulty arises in determining where

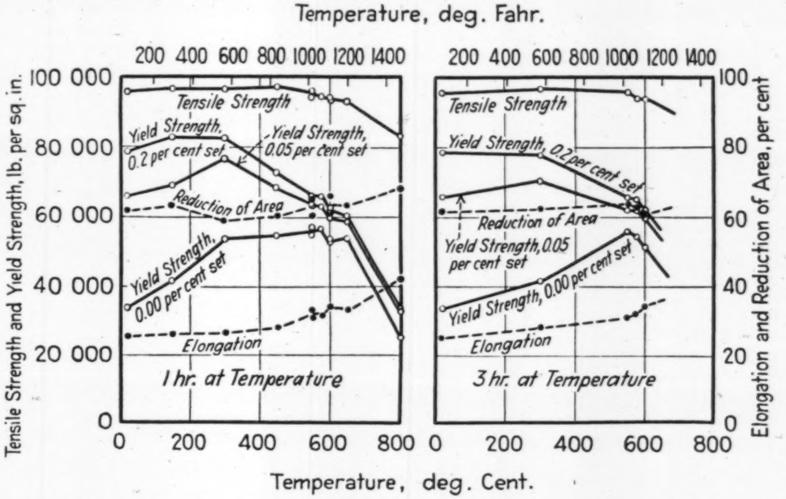

FIG. 2.—Temperature for Stress-Relief Annealing Monel Metal.

½-in. Plate No. 1316-R, Cold-Rolled 10 per cent. 0.357-in diameter bar. 0.00002-in. sensitivity.

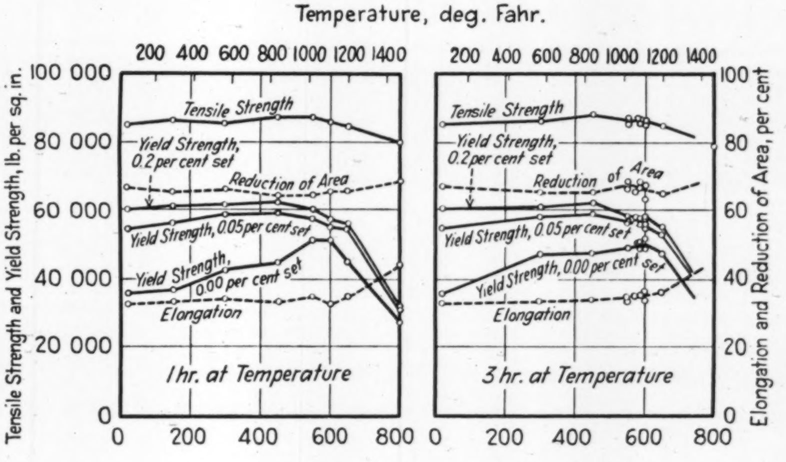

FIG. 3.—Temperature for Stress-Relief Annealing Monel Metal.

½-in. Plate No. 1316-2, Low-temperature Finish. 0.357-in. diameter bar. 0.00002-in. sensitivity.

a straight line matches the stress-strain curves. This has been discussed before.[10,11] But the possibility of using the yield strength, 0.00 per cent

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[10] R. L. Templin, "The Determination and the Significance of the Proportional Limit in the Testing of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 523 (1929).

[11] P. G. McVetty and N. L. Mochel, "The Tensile Properties of Stainless Iron and Other Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Steel Treating, Vol. 11, p. 73 (1927).

set, as a measure of internal stresses has not been extensively discussed. In Table III the effect of locally straining stress-relieved plate is indicated.

The curves in Figs. 2 and 3 and data in Table II show that in the range 550 to 600 C. (1020 to 1110 F.) the maximum increase in yield strength, 0.00 per cent set, is obtained for this high-strength monel metal plate, no matter whether it is slightly cold rolled or hot rolled but finished at a lower temperature than usual. At the higher limit softening is evident. This is an indication of the removal of internal stresses.[5,12] A temperature of 575 C. (1065 F.) therefore appears to be suitable for stress-relief annealing. The change in grain size due to annealing at this temperature is inappreciable. This temperature is not critical; an increase of 25 deg. Cent. (45 deg. Fahr.) or a decrease of even 75 deg. Cent. (135 deg. Fahr.) is not harmful.

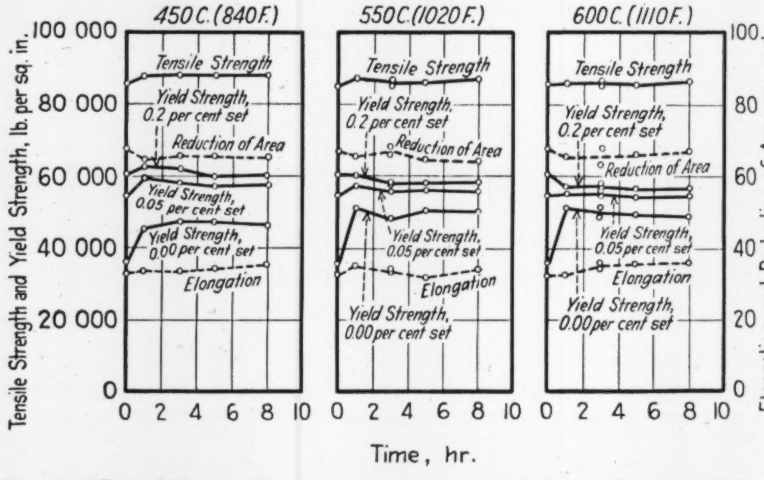

FIG. 4—Effect of Time at Temperature upon Stress-Relief Annealing Monel Metal.
½-in. Plate No. 1316-2. Low-temperature Finish. 0.357-in. diameter bar. 0.00002-in. sensitivity

The effect of annealing upon the yield strength, 0.05 and 0.2 per cent set, is interesting. These strengths are first increased and then decreased. The temperature at which maximum values are obtained is governed by the amount of cold working to which the metal is subjected.

The effect of time at temperature is shown in Fig. 4. The tensile strength reached at 450 C. (840 F.) is higher than at 550 or 600 C. (1020 or 1110 F.). Yield strengths, 0.2 and 0.05 per cent set, pass through slow maxima at 450 C. (840 F.) and sharper maxima at 550 C. (1020 F.). At 600 C. (1110 F.) yield strength, 0.05 per cent set, is unaffected, but yield strength, 0.2 per cent set, is lowered. Undoubtedly if the time at temperature were sufficiently short a maximum value would be observed.

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[12] Z. Jeffries and R. S. Archer, "The Science of Metals," Chapter VI, McGraw-Hill Book Co., Inc., New York City (1924).

TABLE II.—PHYSICAL PROPERTIES OF MONEL METAL PLATE BEFORE AND AFTER ANNEALING.

Property	Before Annealing	After Annealing													
		300C. (570F.)		500C. (930F.)		550C. (1020F.)		575C. (1065F.)		600C. (1110F.)		650C. (1200F.)		800C. (1470F.)
		1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.
PLATE NO. 1316-R, COLD WORKED 10 PER CENT															
Tensile strength, lb. per sq. in.	95 500	96 500	96 500	....	....	95 300	95 500	94 500	94 000	93 800	94 000	93 500	....	84 000
Yield strength, 0.2 per cent set, lb. per sq. in.	79 000	83 000	78 000	....	....	67 000	66 000	66 500	65 500	62 800	62 800	61 000	....	34 000
Yield strength, 0.05 per cent set, lb. per sq. in.	66 300	77 000	71 000	....	....	63 900	63 000	63 500	62 500	60 400	60 000	59 300	....	33 800
Yield strength, 0.00 per cent set, lb. per sq. in.	34 000	54 000	42 000	....	....	56 300	56 000	57 000	55 000	53 500	52 000	54 500	....	26 000
Elongation, per cent	25.7	26.4	28.6	....	....	32.6	31.5	32.2	32.3	34.0	34.3	33.6	....	42.9
Reduction of area, per cent	62.0	59.6	63.0	....	....	63.4	64.4	65.7	62.7	63.8	60.6	63.7	....	69.2
Brinell hardness number, 3000-kg. load	197	202	212	....	....	179	183	170	170	170	170	163	....	121
PLATE NO. 1316-2, LOW-TEMPERATURE FINISH															
Tensile strength, lb. per sq. in.	85 500	86 000	86 000	....	86 000	87 500	86 300	....	86 500	86 000	86 000	85 000	85 000	80 000
Yield strength, 0.2 per cent set, lb. per sq. in.	60 500	62 000	61 000	....	60 000	60 300	58 400	....	57 500	57 200	57 300	55 800	54 800	32 000
Yield strength, 0.05 per cent set, lb. per sq. in.	54 800	58 500	58 000	....	56 500	57 500	55 900	....	55 500	55 000	55 000	54 000	53 000	31 500
Yield strength, 0.00 per cent set, lb. per sq. in.	36 000	43 000	47 000	....	48 000	51 000	48 000	....	50 000	51 000	49 500	45 000	47 000	27 000
Elongation, per cent	32.9	34.3	33.6	....	35.0	35.0	34.0	....	35.0	32.9	35.4	35.0	35.7	44.3
Reduction of area, per cent	67.0	66.3	65.4	....	67.0	65.4	67.1	....	65.7	65.4	65.3	65.4	65.0	68.3
Brinell hardness number, 3000-kg. load	168	160	163	....	163	161	159	....	156	157	158	156	153	118
PLATE NO. 1344-2, LOW-TEMPERATURE FINISH															
Tensile strength, lb. per sq. in.	90 000	....	....	91 000	91 000	90 500	90 500	90 500	91 000	90 000	90 000	89 500	89 000	84 000[a]
Yield strength, 0.2 per cent set, lb. per sq. in.	64 500	....	....	63 500	62 000	62 000	60 000	61 500	61 500	59 500	59 000	57 000	57 000	34 500
Yield strength, 0.05 per cent set, lb. per sq. in.	59 000	....	....	59 500	59 000	59 500	57 000	58 500	58 000	57 000	56 000	55 000	54 500	34 000
Yield strength, 0.00 per cent set, lb. per sq. in.	44 000	....	....	49 000	52 000	53 000	50 000	50 000	52 000	48 000	50 000	50 000	50 000	30 000
Elongation, per cent	34.3	....	....	34.3	34.3	35.0	34.3	34.3	35.7	35.7	35.0	37.2	36.5	44.3
Reduction of area, per cent	64.3	....	....	67.3	67.3	67.3	67.6	66.7	68.3	68.0	68.0	67.0	68.3	69.8
Brinell hardness number, 3000-kg. load	174	....	....	170	170	166	166	166	159	163	159	159	159	128

[a] 2 hr. at temperature instead of 1 hr. at temperature.

TABLE II.—PHYSICAL PROPERTIES OF MONEL METAL PLATE BEFORE AND AFTER ANNEALING (*Continued*).

Property	Before Annealing	After Annealing												
		300C. (570F.)		500C. (930F.)		550C. (1020F.)		575C. (1065F.)		600C. (1110F.)		650C. (1200F.)		800C. (1470F.)
		1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.	3 hr.	1 hr.
PLATE NO. 1521-2, LOW-TEMPERATURE FINISH														
Tensile strength, lb. per sq. in.	86 500	......	......	87 500	87 500	87 500	86 500	87 000	86 000	86 000	86 000	85 000	85 000	......
Yield strength, 0.2 per cent set, lb. per sq. in.	62 500	......	......	61 300	58 500	59 500	57 500	58 500	55 500	56 500	55 000	54 000	51 800	......
Yield strength, 0.05 per cent set, lb. per sq. in.	56 000	......	......	58 000	56 000	57 000	55 000	56 000	53 500	54 000	52 500	52 500	50 000	......
Yield strength, 0.00 per cent set, lb. per sq. in.	39 000	......	......	51 000	46 000	49 000	44 000	48 000	47 000	48 000	45 000	48 000	44 000	......
Elongation, per cent	32.2	......	......	34.3	33.6	34.3	35.0	33.6	35.7	35.0	35.7	37.9	37.2	......
Reduction of area, per cent	65.0	......	......	65.7	68.0	65.0	66.3	64.0	64.7	64.7	65.3	66.5	65.3	......
Brinell hardness number, 3000-kg. load	170	......	......	166	163	163	156	159	149	159	156	156	149	......
PLATE MN, LOW-TEMPERATURE FINISH														
Tensile strength, lb. per sq. in.	93 500	......	94 000	......	95 000	......	......	......	......	......	94 300	......	94 000	88 500[b]
Yield strength, 0.05 per cent set, lb. per sq. in.	56 300	......	57 500	......	60 000	......	......	......	......	......	60 000	......	58 000	34 000
Yield strength, 0.00 per cent set, lb. per sq. in.	33 000	......	50 000	......	55 500	......	......	......	......	......	56 300	......	53 000	27 000
Elongation, per cent	37.9	......	37.9	......	36.5	......	......	......	......	......	37.6	......	37.2	43.6
Reduction of area, per cent	65.0	......	65.0	......	68.6	......	......	......	......	......	69.2	......	65.4	66.3
Brinell hardness number, 3000-kg. load	163	......	174	......	170	......	......	......	......	......	168	......	168	126

[b] 815C. (1500F.) instead of 800C. (1470F.).

The extent of the increase in yield strength, 0.00 per cent set, is dependent upon the temperature and the time at temperature. At 450 C. (840 F.) the maximum increase of 11,000 lb. per sq. in. is reached in 3 to 4 hr.; at 550 and 600 C. (1020 and 1110 F.) the maximum increase of 15,000 lb. per sq. in. is reached in less than 1 hr. After reaching the maximum increase there is a slight tendency for the yield strength to be lowered if held at 600 C. (1110 F.) for periods longer than 1 hr., but there is no such tendency at 550 C. (1020 F.).

The work of Heyn,[4] Jeffries,[12] Grossmann,[13] and of others, suggests a satisfactory basis for an explanation of the observed facts. Heating the strain-hardened metal up to moderate temperatures brings about elastic recovery and the resistance to motion on the planes of slip increases. This is made evident in increased properties such as tensile strength and yield strength. As the per cent set is decreased the yield strength becomes more sensitive to the effect of internal strains which are caused by lack of elastic recovery. Higher temperatures are therefore found to be necessary to complete the process of elastic recovery and so eliminate the last traces of internal strains. As the elastic recovery proceeds the resistance to plastic flow increases, but if and when plastic flow does occur a greater mass movement or slip occurs for any applied stress. This is revealed in a lowering of yield strength at 0.05 and 0.2 per cent set.

TABLE III.—EFFECT OF SENSITIVITY OF EXTENSOMETER UPON PROPERTIES OF ½-IN. MONEL METAL PLATES NOS. 1316-2 AND 1316-R CONTAINING INTERNAL STRESSES AND THE EFFECT OF LOCALLY STRAINING STRESS-RELIEVED METAL.

	Plate No. 1316-2 Longitudinal Low-Temperature Finish			Plate No. 1316-R Longitudinal Cold-Rolled			Plate No. 1316-2 Transverse Low-Temperature Finish		
	Extensometer Sensitivity, 0.0002 in.	Extensometer Sensitivity, 0.00002 in.	Difference	Extensometer Sensitivity, 0.0002 in.	Extensometer Sensitivity, 0.00002 in.	Difference	Extensometer Sensitivity, 0.00002 in.		Difference
							Stress Relieved	Locally Strained After Stress Relieving	
Tensile strength, lb. per sq. in.	85 400	85 500	100	95 400	95 500	100	86 000	86 000	0
Yield strength, 0.2 per cent set, lb. per sq. in.	62 800	60 500	2300	80 500	79 000	1500	57 500	57 800	300
Yield strength, 0.05 per cent set, lb. per sq. in.	58 000	54 800	3200	70 000	66 300	3700	55 300	54 500	800
Yield strength, 0.00 per cent set, lb. per sq. in.	45 000	36 000	9000	39 500	34 000	5500	45 000	39 000	6000
Elongation, per cent	32.9	32.9	0	26.4	25.7	0.7	35.7	35.7	0
Reduction of area, per cent	60.3	67.0	6.7	54.1	62.0	7.9	68.3	66.7	1.6

There are slight discrepancies in the data on hand. They can be explained on the basis that the test specimens were strained by elastic bending during machining operations. It has not yet been so rigorously demonstrated for non-ferrous specimens as it has been for ferrous specimens that machining does not occasionally strain the metal and leave internal stresses in it. The sharpness of the tool and the experience of the operator are factors to be considered. Because of their lower yield strength, 0.00 per cent set, non-ferrous alloys are more prone to be affected by maladjustments of the cutting tool than ferrous alloys.

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[13] M. A. Grossmann and C. C. Snyder, "Hardening by Reheating After Cold Working," *Transactions*, Am. Soc. Steel Treating, Vol. 13, p. 201 (1928).

Summary and Conclusions:

The effect of temperature of annealing and time at temperature upon the physical properties of four heats of high-strength monel metal plate, $\frac{1}{2}$ in. thick, was studied. Particular attention was paid to yield strength, 0.00 per cent set, extensometers of 0.0002 and 0.00002-in. sensitivity being used. In the range 550 to 600 C. (1020 to 1110 F.) the maximum increase in yield strength, 0.00 per cent set, is obtained no matter whether the plate were slightly cold rolled or hot rolled but finished at a low temperature during manufacture. Annealing first increases and then decreases the yield strength at higher per cent sets. The temperature at which maximum values are obtained is governed by the amount of cold working to which the metal was subjected. The tensile strength reached at 450 C. (840 F.) is slightly higher than that at 550 or 600 C. (1020 or 1110 F.). Yield strength, 0.2 and 0.05 per cent set, pass through slow maxima at 450 C. (840 F.) in 2 to 3 hr. and sharper maxima at 550 C. (1020 F.). At 600 C. (1110 F.), yield strength of 0.05 per cent set is unaffected but of 0.2 per cent set is lowered. The extent of increase of yield strength, 0.00 per cent set, is dependent upon the temperature and time at temperature. An increase of 11,000 lb. per sq. in. was noted after 3 to 4 hr. at 450 C. (840 F.), but the maximum increase noted of 15,000 lb. per sq. in. was reached in about 1 hr. at 550 C. (1020 F.) and in less than 1 hr. at 600 C. (1110 F.). If held longer at 550 C. (1020 F.) there is no falling off of yield strength but at 600 C. (1110 F.) there is a very slight falling off. The yield strength, 0.00 per cent set, is sensitive to the presence of internal stresses. When the maximum increase in this yield strength was obtained then it was judged that the internal stresses had been removed, if not completely, then sufficiently for practical purposes. Heating to 575 C. (1065 F.) and holding for 3 hr. and furnace cooling is a suitable treatment for stress-relief annealing this high-strength monel metal plate.

Acknowledgment:

The aid given by all at Watertown Arsenal in carrying out this work is acknowledged. Thanks are given to the International Nickel Co. for supplying the metal for test, to the Ordnance Office for permission to publish and to Col. G. F. Jenks for his active interest and encouragement.

DISCUSSION

Mr. C. A. Crawford[1] (*presented in written form*).—The studies carried out by Mr. Kosting as reported in this paper give an accurate picture of the response of monel metal to stress relief annealing and are of great value to designing engineers who are always interested in the boundary of elastic behavior.

We are accustomed to refer rather loosely to the yield point of a metal, having in mind the highest possible load that the metal will carry without yielding or stretching. We are all aware that the yield point as used in routine testing actually measures a load higher than that at which yielding is incipient. Conscious of this, the designer takes one-quarter or one-third of the "yield point" as the limiting safe stress.

The great sacrifice of useful strength of the metal that is made by such obsolete procedure is adequately shown in Mr. Kosting's data. With carefully determined data on yield strength, at 0.00 set, the engineer can know precisely how far the structure may be loaded before it will permanently change in shape. When at the same time consideration is given to the important increase in proportional limit (preferably as used by Mr. Kosting, "yield strength at 0.00 set") that will result from correct annealing to relieve locked up stresses, the benefits of this paper are at once apparent.

The data make available higher values for safe loading of this important structural material. Mr. Kosting has found the optimum temperature for stress relief annealing monel metal, namely, 575 C. (1065 F.), and his work conclusively shows the merit of applying this stress relief anneal to monel metal structures.

The shape of the curves, Figs. 2, 3 and 4, is particularly interesting. It is a fact that as the amount of cold work increases, the temperature at which relief of stress begins is found to be lower. Photomicrographs indicate that recrystallization through annealing will occur at lower temperatures as cold work is increased. Nonetheless, Mr. Kosting's records show that for both the very slightly cold-worked plate (low-temperature finish) and for that reduced 10 per cent in thickness by cold rolling, the relief anneal at 575 C. (1065 F.) will give the highest values for yield strength at 0.00 set.

This seems important, as for example in the cases of welded assemblies.

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[1] Development and Research Division, The International Nickel Co., Inc., New York City.

The determination of the necessary time-at-temperature is also most helpful.

Every engineer interested in the design and fabrication of strong corrosion-resistant structures will be grateful to Mr. Kosting for this useful paper.

MR. W. A. MUDGE[2] (*presented in written form*).—Tests similar to those reported by Mr. Kosting have been made in our laboratory on the plate materials which Mr. Kosting used, and have duplicated his results within experimental error. His recommended practice is in line with the standard procedure at the Huntington Works of The International Nickel Co., Inc., for stress-relieving high-strength monel metal forgings.

The results obtained with the more sensitive extensometer show that monel metal is an alloy with a reliably true proportional limit (yield strength at 0.00 per cent set) rather than an alloy the stress-strain curve of which tends to curve from its origin, as is generally true for the nickel-copper alloys containing higher percentages of copper. It seems to me that the differences which Mr. Kosting has observed with the two extensometers are not great enough to require the use of the more sensitive instrument for production testing and that this fact can be taken care of in the design safety factor. Additional work will be required to show how much of his observed differences may have been due to straining by elastic bending during machining operations.

TABLE I.—THE EFFECT OF TEMPERATURE UPON STRESS-RELIEF ANNEALING A $1\frac{1}{2}$-IN. DIAMETER COLD-DRAWN MONEL METAL ROD.

Condition, Stress-Relief Annealing Temperature	Yield Strength, lb. per sq. in.			Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
	0.00 per cent set	0.05 per cent set	0.5 per cent elongation under load			
As cold drawn	45 000	84 875	99 425	108 500	20.5	67.3
200 C. (390 F.)	62 500	94 875	103 200	111 200	20.0	65.8
250 C. (480 F.)	67 500	94 000	103 750	113 250	20.0	65.1
300 C. (570 F.)	69 825	93 750	101 150	112 625	20.8	64.4
350 C. (660 F.)	66 500	89 625	97 850	111 575	22.2	65.8
400 C. (750 F.)	60 000	83 125	91 100	110 000	25.2	64.4
450 C. (840 F.)	58 500	78 875	86 925	106 825	26.8	67.8
500 C. (930 F.)	58 000	75 500	81 000	103 350	27.0	69.1
550 C. (1020 F.)	56 125	72 375	77 150	101 250	27.3	68.3
600 C. (1110 F.)	52 500	70 000	73 125	99 250	30.8	71.0
650 C. (1200 F.)	45 000	61 500	66 000	93 125	34.3	71.7
700 C. (1290 F.)	22 500	32 500	34 000	84 625	44.2	73.0
750 C. (1380 F.)	20 000	28 500	33 750	83 625	43.5	73.2

The fact that a slight amount of cold rolling, that is, a maximum of 10 per cent, had no appreciable effect upon the stress-relieving treatment of these hot-rolled plates is of definite practical importance in that the manufacturer can use this finishing operation as a means of more accurate gage control. It has been our experience that Mr. Kosting's findings would not be true if the amount of finishing cold work had been greater, or if the material used had been entirely cold worked, that is cold-drawn rods or cold-rolled strip.

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[2] Works Metallurgist, Huntington Works, The International Nickel Co., Inc., Huntington, W. Va.

Some data on stress-relief annealing of monel metal in cold-drawn rod and cold-rolled strip forms are presented as supplementing the data given in the paper. The accompanying Table I and Fig. 1 give tensile data, averaged to the nearest 25 lb. per sq. in., after stress-relief annealing for 3 hr. at temperatures of 200 to 750 C. (390 to 1380 F.), at intervals of 50 deg. Cent. for a $1\frac{1}{2}$-in. diameter cold-drawn rod. This rod was originally hot rolled at $1\frac{11}{16}$ in. diameter, annealed 45 min. at 820 C. (1505 F.), pickled and cold drawn with two passes, for a total cold reduction of 20 per cent, to $1\frac{1}{2}$ in. diameter. All properties were determined with standard 0.505-in. test specimens, using an Olsen machine of 200,000 lb. capacity and a Ewing extensometer the sensitivity of which was 0.0002 in. In order to preclude any possibility of discrepancies due to straining by elastic bending during machining, all test specimens were machined to size in advance of the stress-relief annealing treatment.

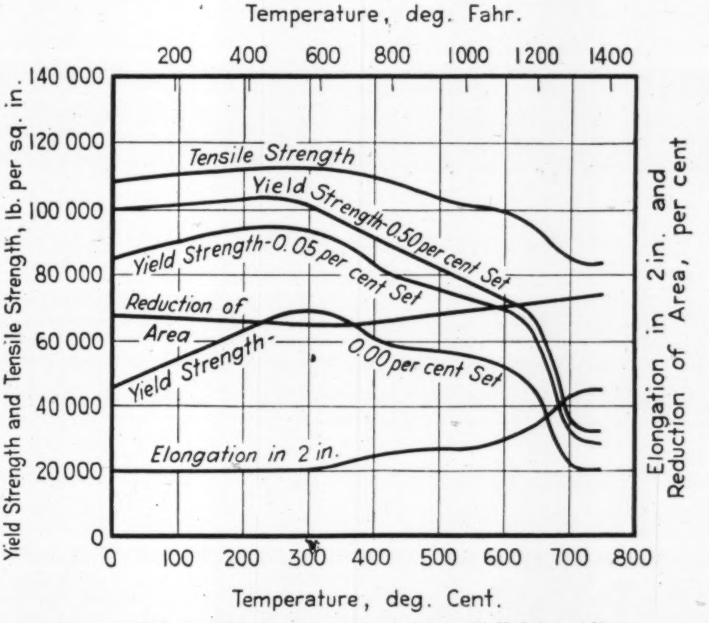

FIG. 1.—Effect of Temperature upon Stress-Relief Annealing.

These figures clearly show that 300 C. (570 F.) is the maximum temperature which may be used to develop the highest properties. In practice we use 275 C. (525 F.), in order to have an operation safety factor. A

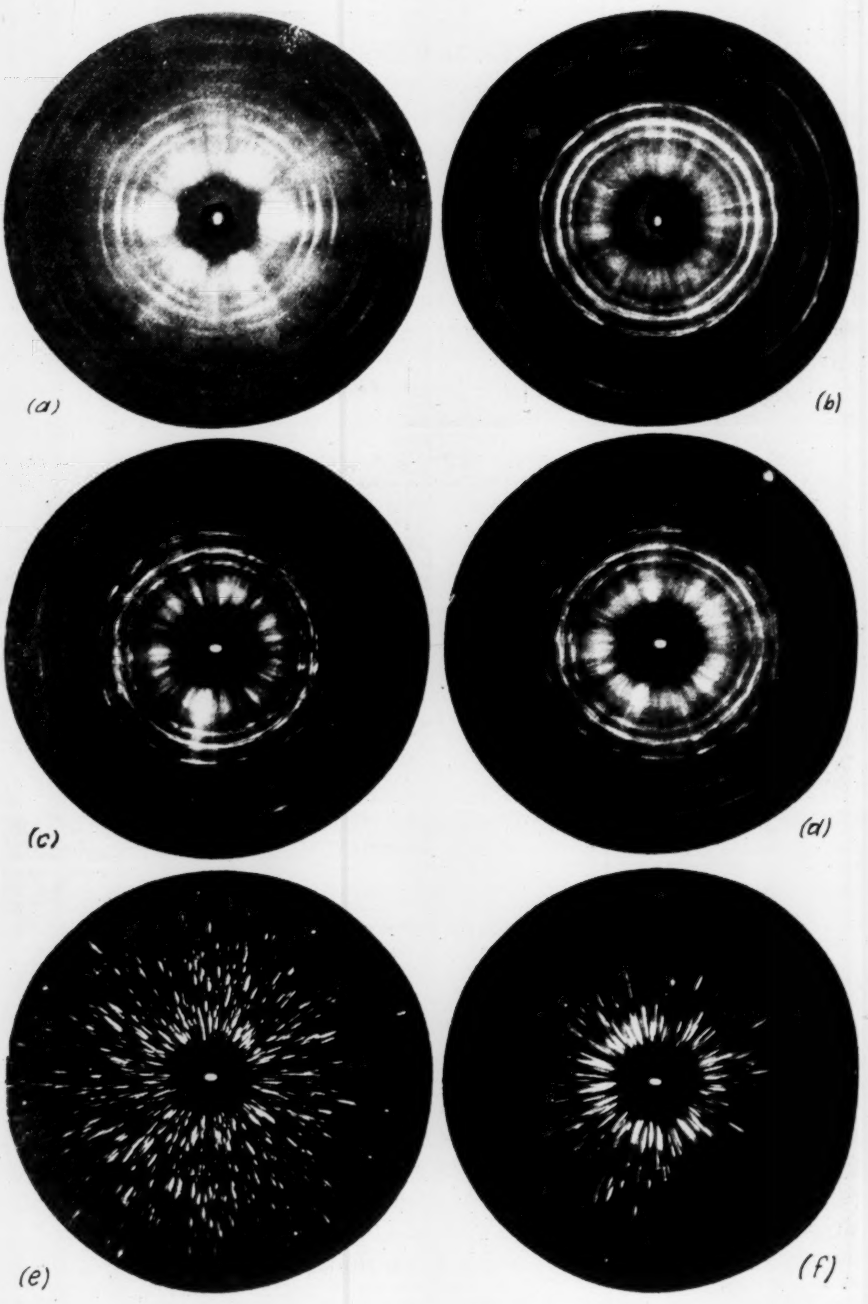

FIG. 2.—X-ray Diffraction Patterns of a 1$\frac{1}{2}$-in. Diameter Stress-Relieved Cold-Drawn Monel Metal Rod. (See Table I.)

(*a*) As cold drawn, 20 per cent reduction, from 1$\frac{11}{16}$ in. diameter.
(*b*) After 3 hr. at 300 C. (570 F.).
(*c*) After 3 hr. at 500 C. (930 F.).
(*d*) After 3 hr. at 600 C. (1110 F.).
(*e*) After 3 hr. at 650 C. (1200 F.).
(*f*) After 3 hr. at 700 C. (1290 F.).

comparison of these results with those given in Figs. 2 and 3, and in Table II of the paper proves my earlier statement regarding the amount of cold working.

It is interesting to follow the progress of this stress-relief annealing by means of X-ray diffraction patterns. These are reproduced in the accompanying Fig. 2. They were made with filed and etched, 0.004 in. thick specimens, polychromatic radiation normal to the direction of cold drawing of the rod, a 0.02-in. slit, a specimen to film distance of 5 cm., and an exposure of 40 hr.

TABLE II.—EFFECT OF TIME AT TEMPERATURE WHEN STRESS-RELIEF ANNEALING A COLD-DRAWN MONEL METAL ROD AT 275 C. (525 F.).

Original cold reduction was 20 per cent.

CONDITION OF MATERIAL	YIELD STRENGTH, 0.00 PER CENT SET, LB. PER SQ. IN.
As drawn	40 000
After 1 hr. at 275 C. (525 F.)	82 000
After 4 hr. at 275 C. (525 F.)	81 000
After 8 hr. at 275 C. (525 F.)	80 000
After 16 hr. at 275 C. (525 F.)	80 000

TABLE III.—THE EFFECT OF TEMPERATURE UPON STRESS-RELIEF ANNEALING COLD-ROLLED MONEL METAL STRIP.

Original hardness before cold rolling—scleroscope 18.

Cold Reduction, per cent	Shore Scleroscope Hardness					
	As Cold Rolled	540 C. (1005 F.)	560 C. (1040 F.)	580 C. (1075 F.)	600 C. (1110 F.)	620 C. (1150 F.)
10	35	28	28	26	25	25
20	41	30	30	29	27	26
30	43	33	32	32	18	18
40	44	34	33	31	18	18
50	45	38	36	28	18	18

Progress in equalization, or relief, of stress is evidenced by a decreasing amount of preferred orientation with increasing temperature up to 650 C. (1200 F.) where recrystallization occurs and random orientation results, although the tensile properties are not yet those of fully annealed material. A temperature of 700 C. (1290 F.) is necessary to accomplish full annealing with some resultant grain growth.

A second example, with cold-drawn rods, shows that there is no appreciable decrease in yield strength at 0.00 per cent set when stress-relief annealing is continued at 275 C. (525 F.) longer than 3 hr. The values are given in the accompanying Table II.

A third example illustrates the effect of stress-relief annealing on heavily cold-worked monel metal. Annealed strip, 0.031 in. thick, was cold-rolled 10 to 50 per cent and heated for 1 hr. at temperatures of 540 to 620 C.

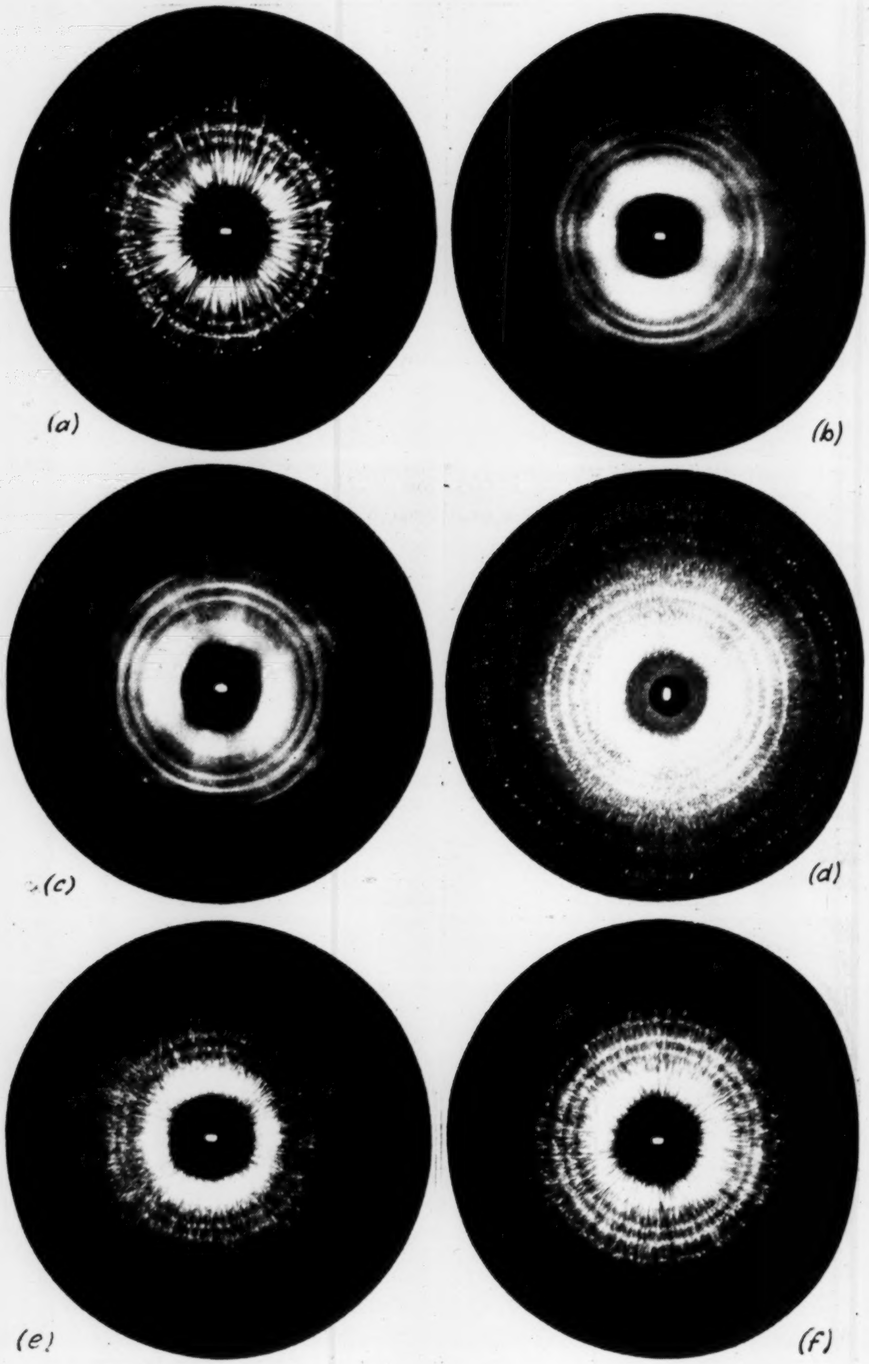

FIG. 3.—X-ray Diffraction Patterns of Stress-Relieved 0.015-in. Cold-Rolled Monel Metal Strip.
(See Table III.)

(*a*) 0.031 in. thick, as annealed 700 C. (1290 F.) for 1 hr., previous to cold rolling, scleroscope 18.
(*b*) As cold-rolled, 50 per cent reduction, to 0.015 in.
(*c*) After 1 hr. at 540 C. (1005 F.).
(*d*) After 1 hr. at 560 C. (1040 F.).
(*e*) After 1 hr. at 580 C. (1075 F.).
(*f*) After 1 hr. at 600 C. (1110 F.).

(1005 to 1150 F.) in steps of 20 deg. Cent. The accompanying Table III and Fig. 3 give a condensed summary of the results.

Here, again, a high temperature of 575 C. (1065 F.) results in partial softening. Cold working progressively lowers the recrystallization temperature of monel metal. And, finally, recrystallization is approximately 40 deg. Cent. ahead of complete softening as indicated by hardness. This is in line with the data for cold-drawn rods.

There are, therefore, two different stress-relief annealing treatments for high-strength monel metal. Monel metal is a solid solution alloy and may be hardened only by cold working or by hot working at a relatively low temperature. This difference in stress-relief annealing treatment depends upon the method used in producing the high strength.

Cold-worked, high-strength monel metal has the best combination of mechanical properties when stress-relief annealed at 275 to 300 C. (525 to 570 F.). Low-temperature finished, high-strength monel metal has the best combination of mechanical properties when stress-relief annealed at 575 C. (1065 F.) as Mr. Kosting shows.

MR. P. H. BRACE.[3]—I should like to ask whether the authors obtained any data as to the magnitude of the residual stresses after their various heat treatments. If we build a structure of this material in which tensile residual stress in one part is balanced by compressional residual stress in another, to what degree will these stresses be eliminated by the strain relieving treatments that have been suggested? The residual stresses in a completed structure are sometimes of considerable engineering importance.

MR. PETER R. KOSTING.[4]—I wish to thank those who have contributed materially to the data given in the paper. In reference to the question raised by Mr. Brace, we have no first-hand information. Initially, an extensive program of study was planned, but when the sensitivity of measurement revealed such marked changes in yield strength, the program was cut short; we are awaiting subsequent developments to reveal the necessity for further study. The point is: is this measure of proportional limit sufficient as a guide in carrying out stress relief annealing practices? Mr. Crawford's discussion stresses what we are doing—using newly developed equipment that is commercially available and time-saving in operation in measuring more accurately the properties of alloys that are useful to the engineer.

(*By letter*).—The contribution of Mr. Mudge enhances the scope of the paper materially. Not only has he checked the original data but extended the data to include material with higher percentages of cold work. The reported sensitivity of his extensometer was the same as used on plate Mn which indicated the same maximum safe temperatures for stress relief annealing as the extensometer of higher sensitivity, thus showing that his information is equally applicable.

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[3] Manager, Metallurgical Division, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[4] Chemical Engineer, Watertown Arsenal, Watertown, Mass.

Experience to date indicates that welded structures are satisfactorily stress relieved by a 3-hr. hold but, if the plate be extensively bent during fabrication, slightly longer holds are desirable.

DUCTILITY TESTING OF ALUMINUM AND ALUMINUM ALLOY SHEET

By R. L. Templin[1]

Synopsis

Because of the great diversion of opinions as to what constitutes ductility of a metal and as to just what measures this same quantity, it has seemed advisable to review a number of existing tests purported to give a measure of this property. This paper makes brief reference to the known effects of the size and shape of test specimen and gage length on the ductility measurements obtained from the tension tests.

The results of bending tests are discussed from the point of view of comparisons with tension test data with evidence to show that there seems to be no reliable correlation between them.

Some results of Erichsen tests are similarly discussed and data presented to show that, here again, there is no reliable correlation between the Erichsen values and tension test values or bending test values.

Results of the Sachs ductility tests have also been discussed with a view to pointing out the difficulties involved in arriving at indices of ductility from this method.

Data from about 1500 observations of these various tests are discussed from the viewpoint that each type of test might offer some particular information regarding a particular type of forming but that no one or any combination of them appears to yield a general quality factor indicating by a single number the quality of the metal in the light of all of the varied definitions of ductility.

The ductility of a metal may be defined as its ability to undergo permanent plastic deformation. When so defined this property of the metal may be considered as synonymous with plasticity. According to this definition of ductility, the forces imposed upon the metal and the stresses resulting therefrom may be tensile, compressive, shearing or any combination of these. A combination of these forces nearly always occurs in the forming, stamping, drawing, pressing, spinning or coining of sheet metals. In an endeavor to select or develop a simple mechanical test for sheet metals, that will readily evaluate their ductility or "workability," many kinds and types of tests have been proposed and used, with only limited success.

Another concept of the ductility of metals is more specifically concerned with their ability to be drawn out, into the form of wire for example, or hammered thin into sheets. Synonymous terms based on this idea are malleability and pliability. While conforming to this idea and yet attempting to be more specific, some authors of materials text books have simply defined ductility as being the final elongation or reduction in cross-sectional area, occurring during the tension test of a metal. When tension tests of thin sheet metals are made in accordance with the standard procedures of this Society, it is necessary to use a specimen of different cross-section for each different thickness of sheet tested. This results in elongation values from a fixed gage length of 2 in. which are not directly comparable because of the effects of total cross-sectional area of specimen on the measured elongations.[2] It would therefore seem necessary to agree upon some one area value to be used in making comparisons based on corrected test results, if elongation values are to be used as a measure of ductility of thin sheet metals. This idea has been suggested by Oliver[3] but apparently not investigated to a sufficient extent to permit of a fair appraisal of its merits.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

The use of the reduction in cross-sectional area as a measure of ductility for thin sheet metals is impractical because of the serious difficulties encountered in the measurement of the dimensions of the fractured specimens.

Martens[4] reported that for many years he had used the ratios of yield strength to tensile strength as criteria of the ductilities of metals, as shown in certain commercial practices, with quite satisfactory results. Reiser[5] in 1881 suggested that the difference between tensile strength and yield strength as well as the elongation might be considered measures of the ductility of metals. Martens later amplified this idea and, considering "toughness" as synonymous with ductility, said that the toughness of a metal was dependent on the ratio of tensile strength to yield strength and the elongation. This relationship may be expressed as follows:

$$\text{Toughness} = \frac{\text{Tensile Strength}}{\text{Yield Strength}} \cdot \frac{\text{Elongation}}{100}$$

He further stated that plasticity was dependent on both "toughness" and yield strength according to the following relationship:

$$\text{Plasticity} = \frac{\text{Toughness}}{\text{Yield Strength}} K$$

where K is simply an arbitrary constant introduced to give results in simple figures. By classifying metals according to these relationships he found that they fell into an order which agreed still better with their ease of working as found by practical experience in contemporary commercial practice.

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[2] R. L. Templin, "Effects of Size ond Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 378 (1926).

[3] D. A. Oliver, "Proposed New Criteria of Ductility from a New Law Connecting the Percentage Elongation with Size of Test Piece," *Proceedings*, Inst. Mechanical Engrs. (London), No. 4, December, 1928, pp. 827–864.

[4] Adolf Martens, "Handbook of Testing Materials," p. 305, John Wiley and Sons, New York City (1899).

[5] F. Reiser, "Das Härten des Stahls in Theorie und Praxis," Arthur Felix, Leipzig (1896).

These indices of ductility, toughness and plasticity, proposed by Martens, offer interesting means for comparing samples from different lots of the same alloy, temper and thickness of sheet metal. In his relationship for toughness and plasticity, however, the previously mentioned effect of specimen size on the elongation factor makes comparisons throughout any appreciable range of thickness rather unsatisfactory. The effects of specimen size and shape on the tensile and yield strengths, however, are negligible when standard specimens are used. The ratio of yield strength to tensile strength, therefore, has been included in the comparisons that will be discussed.

MATERIALS TESTED

For the purpose of comparing the various mechanical properties or relationships that have been suggested as suitable criteria for defining the ductility of metal, the data given in Table I for numerous commercial alloys of aluminum in sheet form will be considered. The alloys given include commercially pure aluminum in three tempers: soft, half-hard and full-hard; the common alloys 3S, 4S and 52S which likewise depend upon definite amounts of cold working after final annealing for their various intermediate and full-hard tempers; and two of the well-known and widely used alloys which depend upon heat treatment for their range of properties. One of these two heat-treatable alloys (51S) requires artificial aging to obtain its maximum tensile and yield strengths, while the other (17S) is the original duralumin alloy which ages spontaneously at room temperature in about four days to attain its optimum properties. The results given in nearly every instance represent the average of four separate tests. Unfortunately, the Erichsen machine available would not test the thicker sheet specimens so some of the values for this test are missing. Likewise the repeated bend testing machine used was not adaptable to the heavier gages available. Chemical compositions for the various alloys are not given because they are within the limits indicated in readily available government specifications. Certain variations in the mechanical properties among the various thicknesses may be noted. With very few exceptions these are in accordance with those variations which may be anticipated in commercial sheet.

DUCTILITY TESTS CONSIDERED

The mechanical property tests which will be considered in attempting to select a suitable measure for ductility include the simple 180-deg. and repeated 90-deg. cold-bend tests, the Erichsen test, the Sachs ductility test and the tension test. The ratio of yield strength to tensile strength will be used and elongation values are also included, despite the limitations pre-

TABLE I.—MECHANICAL PROPERTIES OF VARIOUS ALUMINUM ALLOYS IN THE FORM OF SHEET.

Alloy and Temper	Thickness of Sheet, in.	Tensile Strength, lb. per sq. in.	Yield Strength, 0.2 per cent set, lb. per sq. in.	Elongation in 2 in., per cent	Ratio, Yield Strength to Tensile Strength	Erichsen Value, mm.	Number of Repeated Bends over Radius of $4T^a$		Minimum Diameter for 180-deg. Bend	
							N^a	P^a	N^a	P^a
S-0	1/4	13 630	5 500	38.4	0.40	....	....	....	0*T*	0*T*
	1/8	13 755	4 790	36.3	0.35	15.24	55	54	0*T*	0*T*
	1/16	14 125	5 940	32.9	0.42	11.72	70	57	0*T*	0*T*
	1/32	13 690	5 515	31.3	0.40	10.12	91	77	0*T*	0*T*
	1/64	13 330	4 775	24.4	0.36	8.27	94	93	0*T*	0*T*
2S-½H	1/4	17 895	16 300	14.2	0.91	....	....	....	0*T*	0*T*
	1/8	18 635	17 095	10.6	0.92	....	....	....	0*T*	0*T*
	1/16	19 380	17 725	8.9	0.91	9.51	39	34	0*T*	0*T*
	1/32	18 900	17 200	6.8	0.91	7.68	....	....	0*T*	0*T*
	1/64	19 390	17 065	4.8	0.88	5.00	....	....	0*T*	0*T*
2S-H	1/4	25 115	23 400	11.0	0.93	....	....	....	1*T*	2*T*
	1/8	24 790	22 200	8.0	0.90	10.39	16	16	1*T*	1*T*
	1/16	25 610	22 950	5.9	0.90	8.65	22	18	0*T*	0*T*
	1/32	26 085	23 450	4.3	0.90	6.45	23	20	0*T*	0*T*
	1/64	26 110	23 875	3.5	0.92	5.69	29	26	0*T*	0*T*
3S-0	1/4	15 945	6 640	37.7	0.42	....	....	....	0*T*	0*T*
	1/8	15 705	6 475	37.0	0.41	....	....	....	0*T*	0*T*
	1/16	16 015	6 875	34.2	0.43	11.98	50	44	0*T*	0*T*
	1/32	16 185	6 450	29.7	0.40	10.24	64	62	0*T*	0*T*
	1/64	16 195	5 825	21.9	0.36	8.51	68	65	0*T*	0*T*
3S-½H	1/4	22 195	20 350	12.0	0.92	....	....	....	0*T*	0*T*
	1/8	22 625	20 500	9.3	0.91	....	22	20	0*T*	0*T*
	1/16	22 910	20 450	9.5	0.89	9.70	32	31	0*T*	0*T*
	1/32	22 755	20 500	8.0	0.90	8.03	32	30	0*T*	0*T*
	1/64	23 170	20 900	6.0	0.90	5.78	34	31	0*T*	0*T*
3S-H	1/4	29 945	27 000	7.9	0.90	....	....	....	2*T*	2*T*
	1/8	28 705	25 700	6.8	0.90	....	....	....	1*T*	1*T*
	1/16	29 460	26 000	4.5	0.88	8.44	21	17	0*T*	0*T*
	1/32	29 560	26 325	3.7	0.89	7.12	....	....	0*T*	0*T*
	1/64	30 040	25 900	2.0	0.86	5.87	....	....	0*T*	0*T*
4S-0	1/4	26 715	9 850	22.9	0.37	....	....	....	0*T*	0*T*
	1/8	27 110	9 600	20.2	0.35	11.60	20	19	0*T*	0*T*
	1/16	27 205	10 050	21.3	0.37	9.44	32	31	0*T*	0*T*
	1/32	27 315	9 900	21.1	0.36	8.24	40	34	0*T*	0*T*
4S-½H	1/4	36 005	31 750	8.4	0.88	....	....	....	0*T*	1*T*
	1/8	36 435	32 000	6.5	0.88	....	14	8	1*T*	1*T*
	1/16	35 290	39 500	5.0	0.84	7.70	18	16	0*T*	0*T*
	1/32	36 445	32 000	4.4	0.88	6.91	16	13	0*T*	0*T*
	1/64	36 205	31 500	3.8	0.87	5.84	18	14	0*T*	0*T*
4S-H	1/4	44 360	39 000	6.0	0.88	....	....	....	3*T*	4*T*
	1/8	45 180	39 500	5.1	0.87	....	4	2	4*T*	4*T*
	1/16	45 025	39 250	4.9	0.87	2.73	2	2	3*T*	3*T*
	1/32	46 405	40 500	3.6	0.87	3.71	3	3	3*T*	3*T*
	1/64	44 710	41 250	3.5	0.92	3.53	4	4	2*T*	3*T*
52S-0	1/8	29 980	15 400	25.0	0.51	13.00	27	24	0*T*	0*T*
	1/16	29 030	12 900	23.0	0.44	10.83	28	26	0*T*	0*T*
	1/32	29 190	14 350	23.9	0.49	8.91	33	30	0*T*	0*T*
	1/64	29 450	14 975	21.8	0.51	7.13	42	40	0*T*	0*T*
52S-¼H	1/8	32 885	23 875	14.7	0.73	....	20	15	0*T*	0*T*
	1/16	32 740	23 730	12.7	0.72	8.97	28	23	0*T*	0*T*
	1/32	33 905	25 475	11.2	0.75	6.32	22	20	0*T*	0*T*
	1/64	33 730	25 250	9.9	0.75	5.17	36	24	0*T*	0*T*
52S-½H	1/8	35 265	27 825	11.8	0.79	....	16	12	1*T*	0*T*
	1/16	35 490	27 530	10.5	0.77	8.21	24	20	0*T*	0*T*
	1/32	37 275	30 125	9.5	0.81	6.08	19	17	0*T*	0*T*
	1/64	36 630	29 275	8.2	0.80	4.59	24	18	0*T*	0*T*
52S-¾H	1/8	37 165	31 000	10.2	0.83	....	12	8	2*T*	1*T*
	1/16	39 540	32 260	10.0	0.82	7.83	19	15	0*T*	0*T*
	1/32	40 345	34 075	9.0	0.84	5.45	14	12	0*T*	0*T*
	1/64	39 345	33 150	7.4	0.84	4.51	18	14	0*T*	0*T*
52S-H	1/8	39 375	33 750	9.9	0.86	....	10	6	2*T*	2*T*
	1/16	42 470	36 830	9.1	0.87	6.99	16	11	1*T*	2*T*
	1/32	42 040	36 275	7.9	0.86	4.40	12	10	0*T*	0*T*
	1/64	42 095	36 850	6.9	0.88	4.27	14	9	0*T*	0*T*

TABLE I.—(*Continued*).

Alloy and Temper	Thickness of Sheet, in.	Tensile Strength, lb. per sq. in.	Yield Strength, 0.2 per cent set, lb. per sq. in.	Elongation in 2 in., per cent	Ratio, Yield Strength to Tensile Strength	Erichsen Value, mm.	Number of Repeated Bends over Radius of $4T^a$		Minimum Diameter for 180-deg. Bend	
							N^a	P^a	N^a	P^a
51S-0......	$\frac{1}{4}$........	14 905	5 650	34.7	0.38	....	....	....	$0T$	$0T$
	$\frac{1}{8}$........	14 565	4 940	33.9	0.34	13.56	44	40	$0T$	$0T$
	$\frac{1}{16}$.......	15 365	5 675	31.3	0.37	11.92	57	53	$0T$	$0T$
	$\frac{1}{32}$.......	14 695	5 275	30.4	0.36	10.63	63	60	$0T$	$0T$
	$\frac{1}{64}$.......	14 875	5 575	28.3	0.37	9.32	62	60	$0T$	$0T$
51S-W....	$\frac{1}{4}$........	37 975	25 250	26.8	0.66	....	....	....	$2T$	$2T$
	$\frac{1}{8}$........	38 265	25 450	25.9	0.66	....	9	6	$1T$	$1T$
	$\frac{1}{16}$.......	36 930	22 425	26.2	0.61	9.23	14	13	$1T$	$1T$
	$\frac{1}{32}$.......	38 400	23 800	22.2	0.62	8.50	40	32	$0T$	$0T$
	$\frac{1}{64}$.......	35 815	21 250	23.5	0.59	7.65	44	38	$0T$	$0T$
51S-T.....	$\frac{1}{4}$........	49 240	41 050	14.9	0.83	....	....	....	$7T$	$8T$
	$\frac{1}{8}$........	48 115	41 750	13.9	0.87	....	1	1	$6T$	$6T$
	$\frac{1}{16}$.......	47 910	41 100	12.9	0.86	3.87	2	2	$5T$	$5T$
	$\frac{1}{32}$.......	46 925	39 650	14.0	0.85	5.58	8	4	$3T$	$3T$
	$\frac{1}{64}$.......	47 270	41 150	7.2	0.87	4.31	26	18	$2T$	$2T$
17S-0......	$\frac{1}{4}$........	30 420	20 275^b	16.3	0.67^b	....	....	....	$1T$	$2T$
	$\frac{1}{8}$........	29 315	15 200^b	17.3	0.52^b	10.27	18	16	$1T$	$1T$
	$\frac{1}{16}$.......	29 610	13 250	17.3	0.45	9.33	21	21	$0T$	$0T$
	$\frac{1}{32}$.......	30 560	12 700	18.7	0 42	7.87	37	35	$0T$	$0T$
	$\frac{1}{64}$.......	28 130	11 900	18.1	0.42	7.71	52	47	$0T$	$0T$
17S-T.....	$\frac{1}{4}$........	60 670	39 800	20.0	0.60	....	....	....	$5T$	$6T$
	$\frac{1}{8}$........	61 035	40 000	19.9	0.66	....	4	2	$3T$	$3T$
	$\frac{1}{16}$.......	61 400	38 850	19.0	0.63	6.03	6	4	$3T$	$3T$
	$\frac{1}{32}$.......	61 170	38 150	17.8	0.62	6.92	22	22	$2T$	$3T$
	$\frac{1}{64}$.......	61 895	39 500	16.0	0.64	6.52	45	40	$1T$	$1T$

[a] N = Axis of bend normal to direction of rolling
P = Axis of bend parallel to direction of rolling.
T = Thickness of sheet.
[b] Not fully annealed.

viously noted, because of their widespread use as an approximate measure of ductility.

Cold-bend tests of one form or another have long been used as means for evaluating the ductility of metals. It is not surprising, therefore, that this type of test should have received much attention. It was quite apparent in the early experiments that the results obtained in making the bend test were very much dependent on the methods and apparatus used. A review of the methods and devices in use today for making bend tests of metals reveals a diversity that would permit only general or approximate comparisons. An exception to this statement is the A.S.T.M. Tentative Method of Bend Testing for Ductility of Metals (E 16 – 31 T),[6] which is based on tests of steel and is not applicable to thin-sheet metals less than $\frac{1}{4}$ in. thick.

Recently Schuster,[7] as a result of experiments on steel and wrought iron, concluded that the ductility as shown by bend and tension tests can be correlated with sufficient accuracy for commercial testing. An exception

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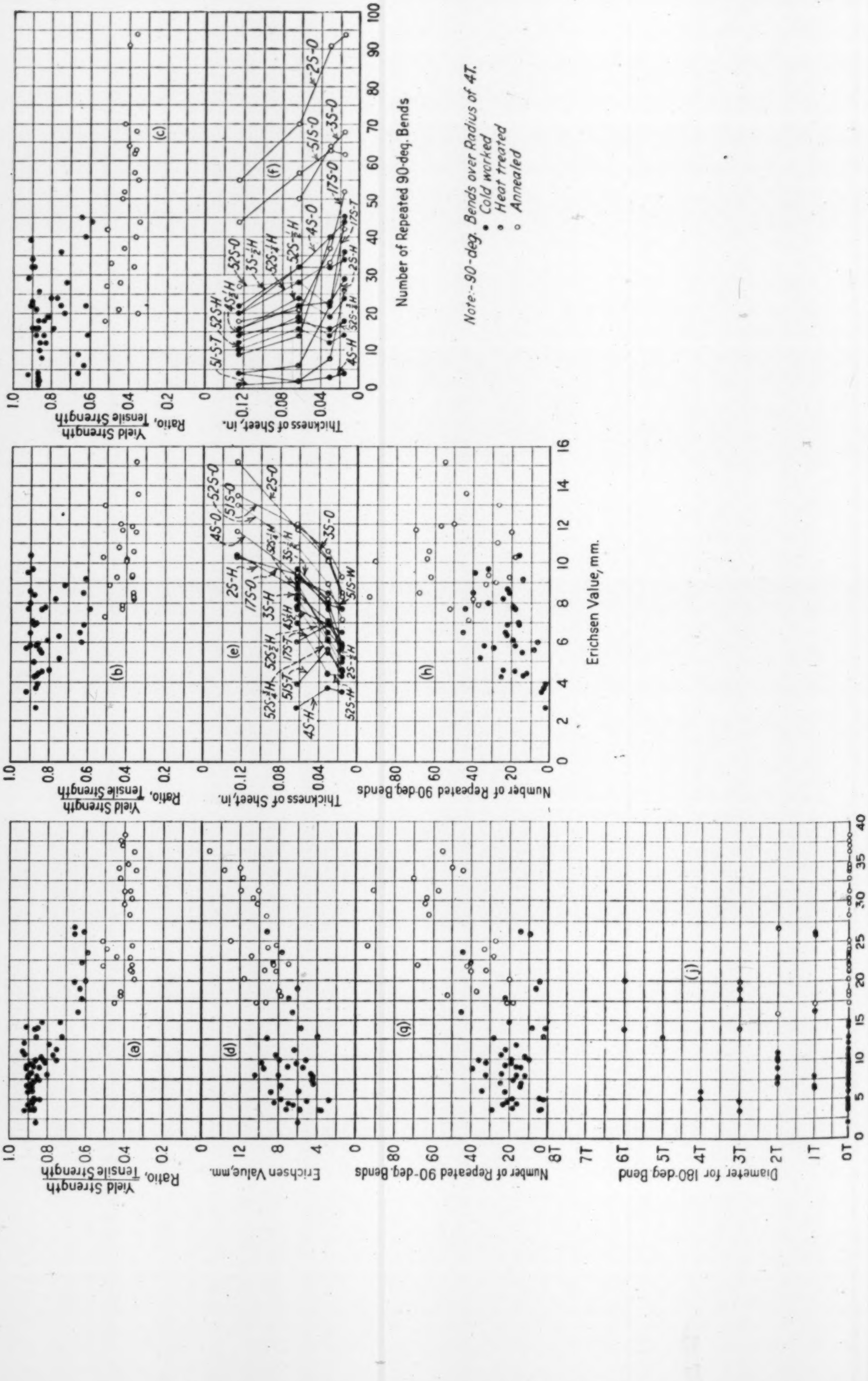
[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 1034 (1931); also 1935 Book of A.S.T.M. Tentative Standards, p. 1366.

[7] L. W. Schuster, "The Bend Test, and Its Value as a Guide to Ductility," *Engineering*, April 5, 1935, p. 372; April 12, 1935, p. 400.


(a)
(b)
(c)
(d)
(e)
(f)
(g)
(h)
(j)
Ratio, Yield Strength / Tensile Strength
Erichsen Value, mm.
Number of Repeated 90-deg. Bends
Diameter for 180-deg Bend
Elongation, per cent
Thickness of Sheet, in.
Erichsen Value, mm.
Number of Repeated 90-deg. Bends
Note:--90-deg. Bends over Radius of 4T.
● Cold worked
● Heat treated
○ Annealed
2S-O
3S-O
51S-O
17S-O
4S-O
52S-O
17S-T
2S-H
4S-H
52S-H
51S-T
51S-W
3S-½H
52S-½H
4S-½H
52S-¼H
52S-¾H
3S-¾H
2S-½H

was reported in the case of steels having low ductility, and numerous other restrictions governing the bend-test procedures seriously limit the scope of the utility of the test. In concluding the discussion of his paper the author made it plain that he preferred the reduction of area in the tension test to the bend test for evaluating ductility.

The repeated bend test has been studied by Van Deusen, Shaw and Davis[8] using various alloys and tempers of brass and nickel-silver sheet. They found that the results obtained depended upon the thickness of the material as well as the qualities of the materials above their elastic limits. They concluded that the repeated bend test offered some promise but more work was necessary to make it available for commercial testing.

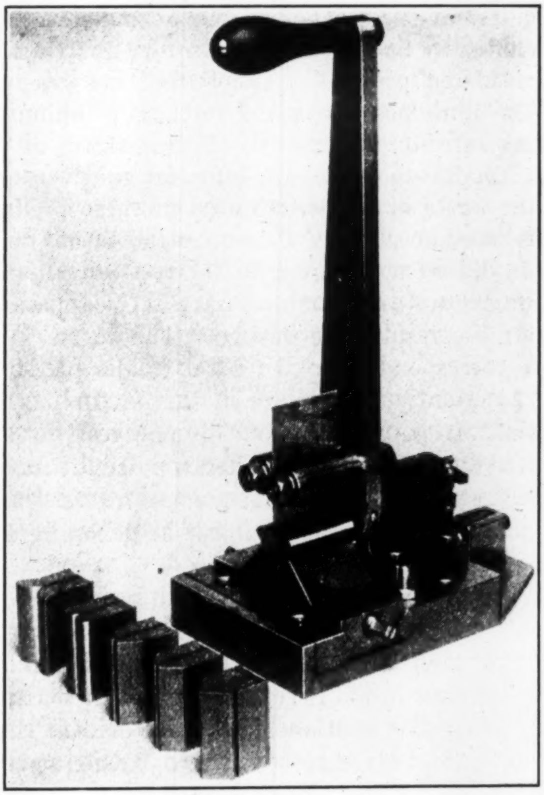


FIG. 2.—Repeated Bend-Test Machine for Thin Sheet Metals.

The bend tests used in obtaining the data given in Table I and plotted in Fig. 1 were of two types. The first was the usual type of simple 180-deg. bend made on a strip sample about $\frac{3}{4}$ in. wide around mandrels of varying diameters. The results are given in terms of the smallest diameter around which the specimen could be bent 180 deg. without visible fracture. In the other type the number of 90-deg. repeated bends over a radius of four times the thickness of the specimen was determined. The machine used in making these tests is shown in Fig. 2.

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[8] H. N. Van Deusen, L. I. Shaw and C. H. Davis, "Physical Properties and Methods of Test for Sheet Brass," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 173 (1927).

The Erichsen test is well known and need not be described here.

A special wedge-drawing apparatus for determining the drawing properties of sheet metals has been proposed and used to some extent by Sachs.[9] Some tests have been reported on zinc and steel sheet, from which it has been concluded by Sachs[10] that "the utility and feasibility of the new test may be considered proved." The author has tried this apparatus experimentally on numerous alloys and tempers of aluminum alloy sheet without obtaining satisfactory results. A comparison of the detailed dimensions of the apparatus used, with information given orally by Sachs, indicates that the angle of the wedge used in these preliminary tests on aluminum alloys was not suitably chosen, so the actual data obtained are not reported. It did seem apparent in the tests on aluminum, however, that certain modifications would be necessary in the apparatus as originally designed, if definitive results are desired. The means provided for adjusting the force corresponding to the blank holder pressure in a drawing operation were apparently inadequate, if this factor has the significance claimed by Sachs. It may be said of this new test, in view of data on aluminum reported by Gillett,[11] that it offers a possible means for evaluating the drawing qualities of sheet metals, but much more experimental work with it would seem necessary before accepting it as generally applicable to all metals.

In Fig. 1 (*a*) it will be seen that, as would be expected, a low ratio of yield strength to tensile strength is accompanied by relatively high elongation and as the ratio of yield strength to tensile strength increases the elongation decreases. This relation is general and is not sharply definitive. It appears, however, that this relationship is more evident than the relationship between the ratio of yield strength to tensile strength and either Erichsen value, Fig. 1 (*b*), or number of repeated bends, Fig. 1 (*c*).

Figure 1 (*e*) and (*f*) show that both the Erichsen value and the number of repeated bends are largely affected by the thickness of the sheet tested. Both of these tests, Fig. 1 (*d*) and (*g*), however, seem to offer more information than the 180-deg. bend, Fig. 1 (*j*), because so many of the alloys and tempers considered bend flat on themselves (180 deg. over zero diameter). The repeated bend test results have been plotted against Erichsen values in Fig. 1 (*h*) to see whether there is any relationship between the two.

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[9] G. Sachs, *Mitteilungen* der deutschen Materialprüfanstalten, Sonderheft 16, p. 11 (1931).

W. Riem, "Untersuchungen über Tiefziehen," *Zeitschrift für Metallkunde*, Vol. 24, No. 7, July, 1932, p. 157.

[10] G. Sachs, "New Researches on the Drawing of Cylindrical Shells," *Proceedings*, Inst. Automobile Engrs. (British), p. 588 (1934–1935).

[11] H. W. Gillett, "Testing Deep Drawing Qualities of Sheet Metals," *Metals and Alloys*, Vol. II, p. 214 (1931)

While the Erichsen value increases as the number of repeated bends increases, in a general way, it is evident that the relationship is not definite.

Attempts to correlate Erichsen test values with commercial fabrication operations such as drawing, spinning, pressing, etc., have not yielded satisfactory results in the case of any aluminum alloy sheet. This experience agrees with the facts brought out in a recent symposium on "Cold Pressing and Drawing" presented before the British Institution of Automobile Engineers,[12] although Graves[13] reported favorably on this test as a measure of the drawing qualities of steel sheet used in automobile bodies.

The various alloys and tempers of materials listed in Table I, when compared on the basis of the ratio of yield strength to tensile strength, will be found to fall into an order which agrees reasonably well with their relative workabilities. This ratio is especially sensitive for the annealed or "dead soft" temper of the materials, as has been pointed out elsewhere.[14] It is also somewhat definitive when determining the relative workability of the harder tempers of the heat-treatable alloys, but not satisfactory as a criterion for discriminating between the harder tempers of the cold-worked alloys. The ratio values for 51S-W and 17S-T, for example, are about the same, and experience has shown that these materials have about the same forming characteristics. On the other hand, the ratio values for 2S-½H, 2S-H, 3S-½H, 3S-H, 4S-½H, 4S-H, 52S-H and 51S-T are all high, ranging from about 0.85 to 0.93, yet there are pronounced differences in their relative workabilities as suggested by their respective repeated bend values and actually found in commercial experience. Furthermore, it has recently been shown by Guyer and Varley[15] that commercially pure aluminum in the intermediate tempers, similar to 2S-½H, will withstand more drawing than when in the fully annealed temper, 2S-O.

In summarizing the foregoing it may be pointed out that many attempts have been made to devise or select a mechanical test which will more adequately define the plastic properties of sheet metals in the hope that such a test may be used as a reliable index of the workability of the materials, with particular reference to forming, stamping, drawing, pressing, spinning and coining. While there is some confusion in the definition of ductility as a property of metals, yet this property is generally considered the most desirable one to be determined when attempting to evaluate workability. All of the measures of ductility considered have been shown to possess serious limitations when applied to aluminum alloy sheet of various alloys and tempers throughout a commercial range of thickness.

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[12] "Cold Pressing and Drawing" (Symposium), *Proceedings*, Inst. Automobile Engrs. (British), pp. 543–630 (1934–1935).

[13] W. H. Graves, "Sheet Steels for Automobiles," Symposium on Developments in Automotive Materials, Detroit Regional Meeting, Am. Soc. Testing Mats., p. 18 (1930). (Available as separate publication.)

[14] R. L. Templin, "Effects of Cold Working on Physical Properties of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., p. 466 (1930).

[15] A. G. C. Guyer and P. C. Varley, "A Deep Drawing Test for Aluminum," *Journal*, Inst. Metals (British), Vol. 3, Part 1, January, 1936, p. 7.

While many years of experience in the production of commercial aluminum sheet have demonstrated that some of the tests considered can be and are used to control uniformity of product, yet they are not sufficiently definitive for determining slight differences in workability. Perhaps this is not surprising when consideration is given to the many factors, other than ductility, which enter into nearly all sheet-metal working operations.

Acknowledgment.—The author wishes to acknowledge the assistance of Messrs. R. G. Sturm, J. O. Lyst and C. Dumont, members of the Aluminum Research Laboratories, in the obtaining and preparation of the data used.

FLEXURE AND TORSION TESTING OF COPPER WIRE

By S. E. Borgeson[1]

Synopsis

This paper discusses the mechanical properties required by copper magnet wire and analyzes the various tests used to measure these. It is demonstrated that the torsion test permits the properties to be expressed in conventional units instead of arbitrary scale units. A simple type of direct reading torsion tester is described and a comparison is given of the results of tests by torsion and other methods.

Introduction

It has become increasingly evident in recent years that the usual tests for tensile strength and elongation do not give sufficient information of the mechanical properties of wires for their successful application to many industrial uses. Among these are the use of soft copper wire in the winding of coils and magnets. Although very little has appeared in the technical literature on the subject, the number of new types of testing machines that have appeared give an indication that the need for additional tests is recognized.

The experiences of manufacturers and users of wire indicate that tensile strength and elongation tests fail in three respects:

1. They are not sufficiently sensitive to slight variations in the hardness of soft wire.
2. They are inconsistent with other tests for such purposes as the determination of optimum annealing temperatures, and
3. They do not show the effect of differential work-hardening of different parts of the wire due to flexure.

Barker and Bailey[2] have shown how incomplete is the correlation of tensile strength and elongation tests, not only with hardness and flexure tests, but also with grain size.

In order to find a satisfactory method of testing soft copper wire to determine its suitability for various industrial applications, an investigation was made of some of the types of tests which have been proposed. This investigation revealed some of the defects of the proposed tests and led to the development of another type of test which is described.

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[1] Mechanical Engineer, General Cable Research Laboratories, Rome, N. Y.

[2] L. B. Barker and C. A. Bailey, "The Effect on the Physical Properties of Bending Copper Wire of Different Grain Sizes," *Wire and Wire Products,* September 1935, p. 375.

SOME MECHANICAL PROPERTIES OF WIRE

An instance of the need for further tests to determine the mechanical properties of wire is furnished by the service requirements of copper magnet wire. The qualities desired in magnet wire differ, depending upon the size of the wire. In the finer sizes of wire, those smaller than about No. 24 A.w.g. (0.0201 in. in diameter) a high value of toughness is required, for they are often stressed almost to the point of rupture during manufacturing operations. Ultimate toughness, which is measured by the energy required to rupture, may be indicated by tension and elongation tests, or tested directly by means of a testing machine of the pendulum-impact type. In this type of tester the wire is ruptured by the drop of a pendulum, and the energy consumed is read directly in inch-pounds per inch. This may then be converted to inch-pounds per cubic inch by dividing by the cross-sectional area.

Coil winding and similar applications require magnet wire in the heavier sizes to be dead-soft, that is, plastic, in order that it may retain any form it is given. The ratio of elastic to total deformation in magnet wire is termed its "springiness" or "spring-back." It may either be measured directly as a ratio, usually in terms of some arbitrary unit, or indicated by a hardness test. For, since the modulus of elasticity in copper changes very little with cold-working, this ratio is approximately proportional to the hardness.

Hardness refers to resistance to deformation. It is usually measured either by the force required to produce a given deformation, or in terms of the amount of deformation produced by a known force. Hardness tests differ according to the type of deformation produced, the choice depending upon the manner in which the material tested is to be used. The value of a hardness test depends upon both the type of deformation and the accuracy with which it is measured.

In the case of magnet wire, the hardness is not necessarily uniform over the cross-section. In wire which has been bent over sheaves, for example, the hardness of the wire over its cross-section varies as a function of the distance from the neutral bending axis. The resistance to flexure of such wire is greater than the resistance as computed from the average value of the hardness over the cross-section as determined for example by tension tests. Although for practically all applications of soft wire a test is desired that will indicate the resistance of the wire to flexure, it is obviously impractical, except in the case of a rectangular wire, to specify the resistance to flexure in terms of a definite neutral surface. The test should rather indicate the average resistance to flexure for all bending planes. The determination of a hardness value of a wire which will indicate its resistance to flexure is therefore limited to flexure and torsion tests in which the deformation is a function of the radial distance from the wire axis.

FLEXURE TESTS

The principal types of flexure tests are the "springiness" types and the cantilever types. "Springiness" tests are made by bending the wire specimen to a given radius of curvature and then releasing one end. The angle through which the free end springs back, gives the measure of the wire hardness. The units of measurement are arbitrary and unless the radius of curvature is made proportional to the wire diameter, the results on various sizes of wire are not directly comparable. The effect that is measured is small compared to the length of the specimen and the test result is subject to variations depending upon the method of bending and the direction of previous bends in the wire.

Cantilever beam flexure tests are made by holding one end of the wire specimen rigidly and deflecting the other end by some means. In these tests the strain on the wire is not directly measurable as it varies with the hardness of the wire. For small deformations the maximum strain is given approximately by the following formula:[3]

$$\delta_{\text{max.}} = \frac{d}{L}\left(\frac{M}{2}\frac{d\theta}{dM} + \theta\right)\dots\dots\dots\dots\dots\dots\dots(1)$$

where d = the wire diameter,
L = the effective length of the specimen,
M = the bending moment, and
θ = the angular deflection of the end of the wire in radians.

The function $\frac{d\theta}{dM}$, and therefore the strain, increases with the hardness of the wire. For this reason, with a given angular deflection of the end of the specimen, the harder of two wires is tested at the greater maximum strain. The harder the wire, the greater is the concentration of the strain. The test values, therefore, increase more rapidly with hardness than if the values are determined at a fixed radius of curvature.

On the other hand, when a constant deflecting force is used, the deflection as measured on a soft wire is obviously greater for a given maximum strain than that of a hard wire.

If the effective length of the wire specimen in a cantilever test is kept constant, the strains will vary also with the wire diameter. The test values on wires differing in diameter are therefore not comparable. This makes it necessary to set separate hardness standards for each size of wire.

All flexure tests were found to give somewhat erratic results depending upon the direction in which the wire has previously been bent compared to the plane of bending in the tester

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[3] This equation is obtained by transposing the equation for the deflection of a cantilever beam of plastic material.

In one type of test studied, the wire specimen is bent by a pendulum dropped from a predetermined height and the angle of deflection is measured. Since the energy imparted by the pendulum is proportional to the difference between the height of fall and height of recovery of the pendulum, and the height of recovery depends on the angle of deflection of the wire, both the maximum deformation and the deflecting force vary with the hardness of the wire.

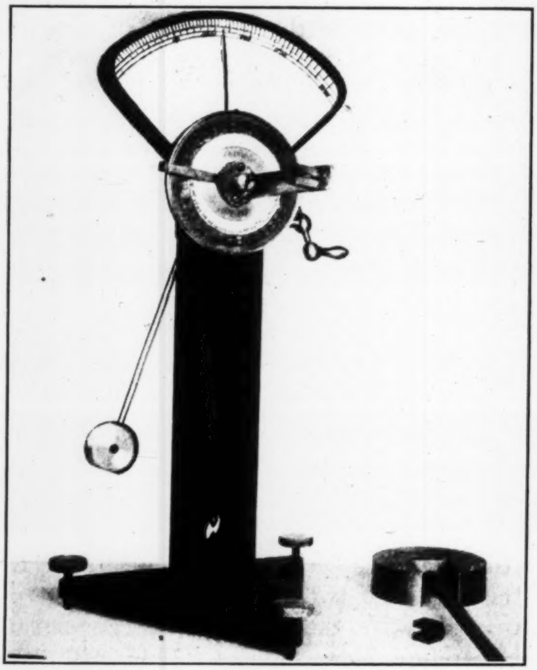

FIG. 1.—Experimental Torsion-Testing Machine.

For mechanical similarity in flexure tests the effective length of the specimen should be proportional to the wire diameter. With a given angular deflection, the strain then depends only on the hardness of the wire and not upon the wire diameter. The hardness value can then be given in terms of the deflecting force divided by the cross-sectional area of the wire, making the hardness values on wires of different diameters comparable. This type of test is also suited to rectangular wire for which more accurate tests are not available. The specimen length is then made proportional to the thickness of the wire.

TORSION TESTS

Emmons[4] lists a number of the advantages of the torsion test for the study of tool steels. The torsion test has also a number of advantages for the testing of wire. Among these are the following: Small deformations can be accurately measured; the deformation is spread uniformly over a greater length of the specimen and the effects of localized stresses are reduced; the maximum strains on the various wire sizes can be made equal and the test results can be compared directly; the strain in the wire is proportional to the distance from the wire axis and torsion tests should, therefore, indicate the effects of work-hardening by flexure; and the test results can be given in terms which are independent of the testing machine used. This is of value since a number of types of torsion testers are available.

Although the maximum stress in torsion cannot be measured directly, the data of torsion tests can be conveniently expressed in terms of the mean stress as defined by the formula:

$$\text{Mean stress} = S_m = \frac{3}{2\pi a^3} T \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where a is the radius of the wire and T is the torque. If T is measured in inch-pounds and a in inches, the mean stress is given in pounds per square inch. The mean stress, therefore, is the stress which if uniformly distributed throughout the wire would give the same torque as the actual stresses. Recording the data in terms of mean stress not only permits direct comparison of data on different wire sizes but also simplifies the computation of the maximum stress where this is required. The maximum stress can be obtained graphically from the mean stress data by means of the equation:[5]

$$\tau_{\text{max.}} = \frac{\theta}{3} \frac{dS_m}{d\theta} + S_m \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

where θ is the angle of twist.

DESIGN OF A TORSION TESTER

In order to determine the value of the torsion test as a measure of the hardness of wire, the experimental tester shown in Fig. 1. was designed. This was designed for wire sizes from Nos. 14 to 24 A.w.g.

Description of Tester:

One end of the wire specimen is rotated by means of a crank operating through a worm and worm gear. The angular rotation of the wire is measured by a scale calibrated in circular degrees. The other end of the wire is attached to a shaft carrying a torque beam, and a pointer which moves across a scale calibrated in pounds per square inch of mean stress.

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[4] J. V. Emmons, "Some Physical Properties of Hardened Tool Steel," *Proceedings*, Am Soc. Testing Mats., Vol. 31, Part II, p. 47 (1931).

[5] This is obtained by transposing the equation for the torque required for twisting a plastic cylinder and substituting the value of T from Eq. 2. See A. Nadai, "Plasticity," McGraw-Hill Book Co., Inc., New York City (1931).

The weight of the beam is 0.220 lb. Its lever arm is adjustable to lengths proportional to the cubes of the diameters of the wires to be tested. Where a is the radius of the wire to be tested, the lever arm of the torque beam is set at 200,000 a^3 in., giving a maximum torque of 14,000 πa^3 in-lb. The full scale reading is therefore, 21,000 lb. per sq. in. since the mean stress is:

$$S_m = \frac{3}{2\pi a^3} T \qquad (4)$$

Wire Specimen:

It was found that if a soft wire is given a sharp bend and then tested, the torsion strains are not carried beyond the beginning of the curvature of the bend because of hardening by flexure. For this reason it was decided to give the wire specimen a sharp bend at each end to limit the length in which strain takes place. The specimen length is kept constant by bending it in a clamp. The length of the specimen used in the test was 3 in.

Operation of Tester:

To operate the tester, the torque beam is adjusted to the position indicated for the wire size to be tested. The wire is placed in the specimen slots and the circular degree scale is adjusted for zero reading. The crank is then turned at constant speed through an angle corresponding to the desired strain. The mean torsional stress is then read directly.

Because the average service condition requires a maximum deformation of wire of about 2 per cent, torsion tests were made at an angle of twist of 0.04 radians, which corresponds to approximately 2 per cent maximum strain. For a 3-in. sample, the rotation in circular degrees was about 13.75 deg. multiplied by the reciprocal of the wire diameter. For example, in making a test on a No. 14 A.w.g. (0.06408 in. in diameter) wire, the lever arm is set at 200,000 × 0.03204^3 in. = 6.578 in. Since 13.75 deg. ÷ 0.06408 = 215 deg., the wire is turned through an angle of 215 deg. and the mean stress is read from the scale.

TEST DATA

Several hundred samples have been tested on the torsion tester and comparative results using the torsion tester, three types of bend testers and tension and elongation tests have also been obtained. Tests were made on tough pitch electrolytic and oxygen-free copper under various

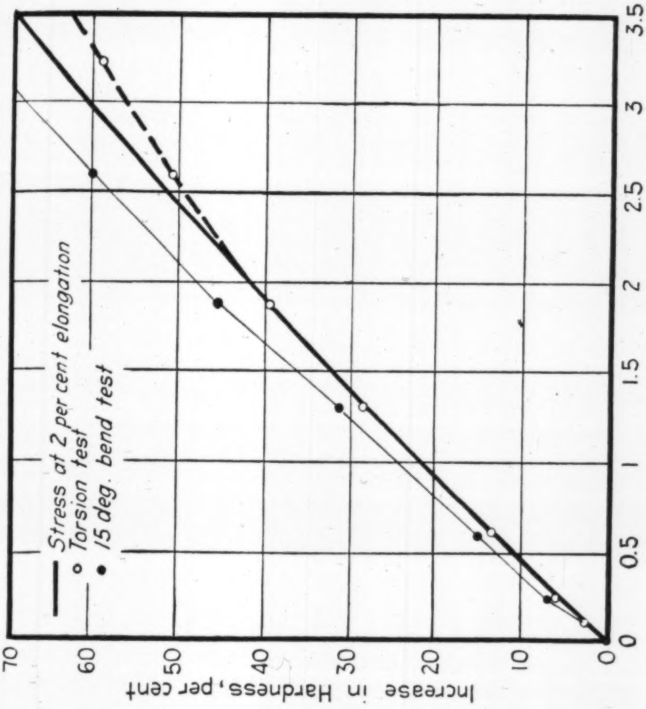

FIG. 3.—Comparison of the Results of Tests Made by Torsion, Flexure, and Elongation on Copper Wire of Varying Hardness.

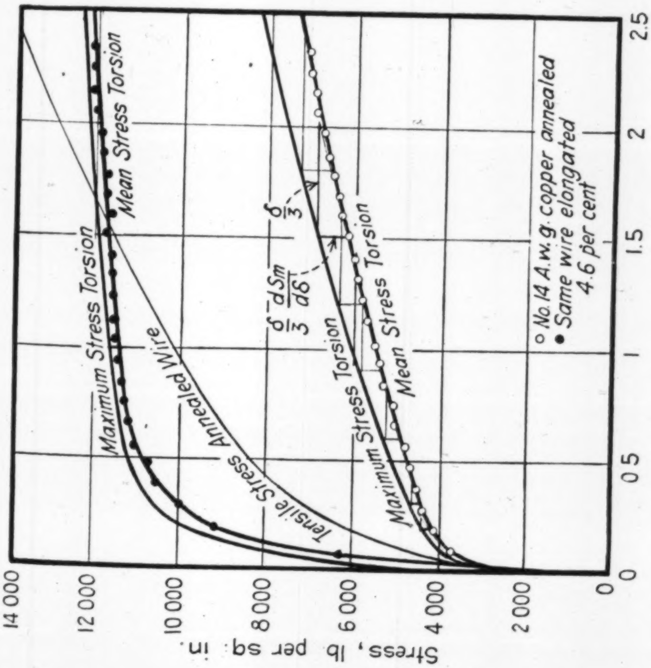

FIG. 2.—Typical Tension and Torsion Stress-Strain Curves of Copper Wire Showing Method of Obtaining Maximum Stress.

annealing conditions and after various shop operations which effected the wire hardness. Tests have been made on both bare and insulated wire.

To illustrate the use of the torsion-testing machine and give some comparison with other methods of testing, a few typical tests are presented below.

The material used for these tests was tough pitch electrolytic copper wire drawn from 0.25 in. rod and annealed for 2 hr. at 600 C. in straightened lengths in order to prevent any hardening due to flexure. The wire on which tests were made included about fifty lengths each of Nos. 11, 14, 18, and 23 A.w.g. The curves shown were made from test data on No. 14 A.w.g. (0.06408 in. in diameter) wire with the exception of the one showing the effect of annealing temperatures which was made from data on 0.091-in. diameter wire.

Figure 2 illustrates the typical stress-strain curves in torsion and the variation in the form of the curve with the hardness of the wire. One of the curves was made from data on wire as annealed, another from data on a wire elongated 4.6 per cent. For comparison a tensile stress-strain curve on the annealed wire is plotted. The method of obtaining the maximum stress curve is illustrated. The points plotted illustrate the consistency of the results obtained by the torsion test with comparatively simple and inexpensive apparatus.

The other three curves illustrate tests on wire in which the hardness has been changed in various ways. Figure 3 shows points plotted from data on wire hardened by various amounts of elongation. The torsion data are shown compared with tension data made by measuring the wire samples with an additional 2 per cent deformation and with data made by means of a cantilever type of test. The hardness as shown by torsion tests parallels that measured by the tension test up to 2.5 per cent elongation. Beyond this point the torsion test results are lower, probably due to directional properties in the wire. The curve plotted from the cantilever flexure test shows a greater increase in hardness because of the increase in the strain with the wire hardness, which is typical of this type of test. The data are plotted in percentage of that on the annealed wire because the numerical values obtained in the cantilever flexure test depend upon the dimensions of the testing machine used.

The effect of differential hardening by flexure is illustrated in Fig. 4. Wires were bent around various sizes of mandrels and straightened. The abscissa values are given as the ratio of the wire diameter to the diameter of curvature, this ratio being in proportion to the maximum strain producing the work-hardening. The cantilever flexure tests are somewhat erratic because the plane of the bend during the test did not coincide with the previous bend. The total elongation data illustrates the lack of sensitivity of this test. The tensile strength of the wire specimens remained constant within the limits of error of the tension testing machine used.

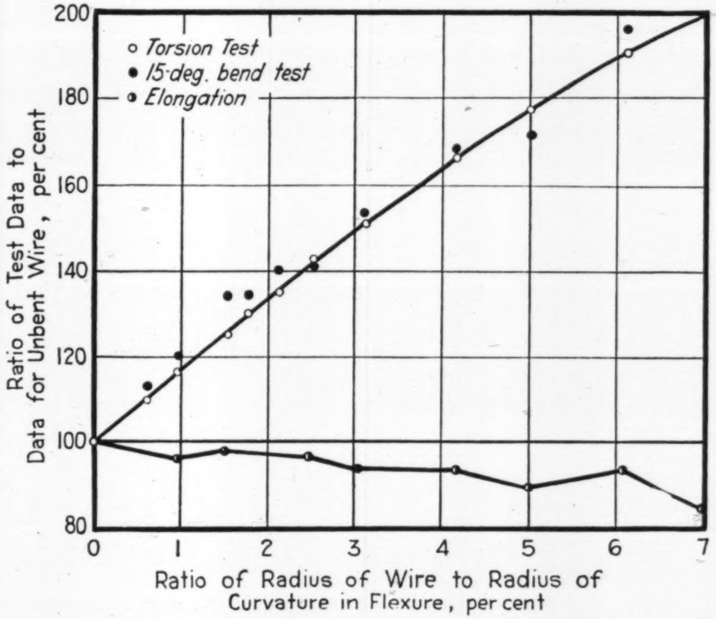

FIG. 4.—Comparison of the Results of Tests on Copper Wire Made by Torsion and Flexure Compared with Total Elongation Values.

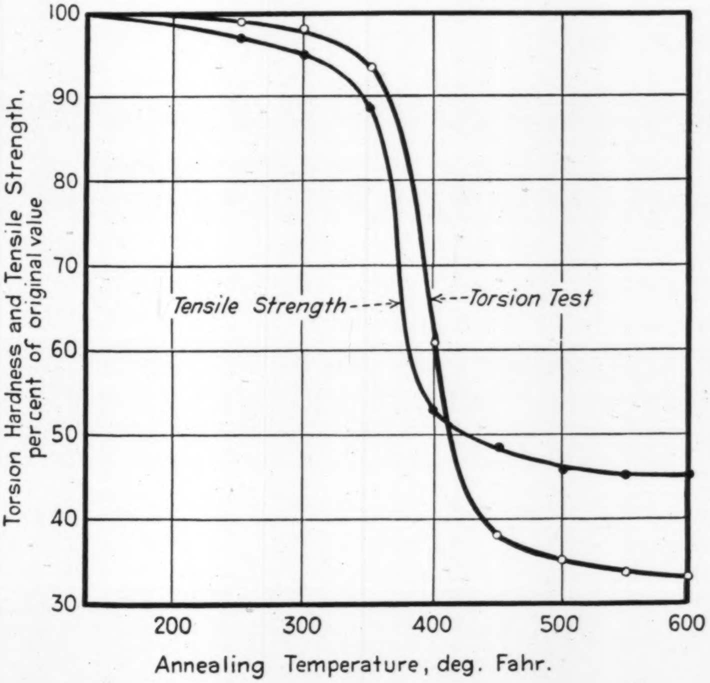

FIG. 5.—Tensile Strength and Torsion Test Results at Various Annealing Temperatures.

Figure 5 is plotted from tests on specimens of 0.091-in. diameter wire drawn from 0.25-in. rod and annealed for ½ hr. at the temperature shown. The curve shows the greater sensitivity of the torsion test in the case of the annealed wire. The minimum hardness as shown by the torsion test is obtained at higher annealing temperatures than the minimum as measured by tensile strength.

CONCLUSIONS

It is felt that the torsion test offers a consistent and accurate method of determining the hardness of wire, especially of soft wire for which tension and elongation tests are not sufficiently sensitive. It gives results which are consistent with service applications in which the resistance of the wire to flexure is the factor determining its usefulness and enables the hardness of the wire to be specified with reference to a specific testing machine.

As a means of studying the effect of annealing schedules and manufacturing processes on wire, the torsion test has a number of advantages including uniform distribution of strains over the length of the test specimen, the possibility of accurate measurement of the strains, and ease and simplicity in operation.

Acknowledgment.—I am indebted to the General Cable Corp. for permission to publish the above and to co-workers in its research laboratories for assistance in its preparation.

DISCUSSION

MR. SAM TOUR.[1]—The paper by Mr. Borgeson is worthy of much detailed study and the author is to be complimented for the excellent work he has done in the development of his torsion test and his torsion testing machine. However, all the statements made need not be fully agreed with, and particularly the somewhat disparaging remarks with reference to flexure testing may well be challenged.

The author rightfully points out that tension and elongation tests do not show the effect of differential work-hardening of different parts of the wire due to flexure. But he then advocates a torsion test which has this same failing. He critizes all flexure tests because he found they gave results depending upon the direction in which the wire had been bent, and further states that "it is impractical, except in the case of rectangular wire, to specify the resistance to flexure in terms of a definite neutral surface."

There is nothing impractical in specifying the resistance to flexure in terms of the plane of bending of the wire when removed from a spool, reel or coil. It is being done regularly and is proving a satisfactory method as indicated by the proposed methods for testing being recommended by the Society's Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys.[2]

In connection with flexure tests, the author calls attention to the fact that "for mechanical similarity in flexure tests the effective length of the specimen should be proportional to the wire diameter," yet in the proposed torsion tests the lever arm of the machine must be adjusted each time to a length proportional to the *cube* of the diameter of the wire to be tested. To adjust the effective length of the specimen proportional to its diameter would seem to be just as simple as to adjust the lever arm of the machine proportional to the cube of the test wire diameter.

It seems that the author of the paper finds objection to the bend test due to having made crude bend tests and getting erratic results, and not because the bend test inherently does give such erratic results. A bend test properly carried out gives just as smooth, uniform and nice a curve as is shown for torsion tests.

The description and photograph of the testing machine used does not make clear what, if any, arrangement is provided to take up the length of the test specimen during the application of torsion. It seems that this should be taken into consideration.

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[1] Vice-President, Lucius Pitkin, Inc., New York City.

[2] Proposed Method for the Bend Testing of Wire, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 162 (1936).

The author has elected to compare materials by comparing the load applied for a constant deformation of 2 per cent. In order to arrive at this constant deformation the total angle of twist would presumably be different for each wire diameter being tested. This total strain of 2 per cent actually represents a very considerable deformation, as shown by Fig. 2 of the paper where the upper curve has practically flattened out while the lower curve continues to ascend. If one elected to compare the materials at 1 per cent strain, the ratio would be 2 to 1 whereas at 2 per cent strain the ratio is about 3 to 2.

It is often possible to select the method of interpreting curves so as to give values of large spread. Such large spreads do not always mean greater sensitivity of the test. For example, in Fig. 2 of the author's paper, if one elected to compare the wires on the basis of percentage of strain for a given load of 6000 lb. per sq. in., a ratio of about 1 to 30 would result. For a load of 4000 lb. per sq. in., a ratio of only about 1 to 4 would result. If large ratios are desired, the proper conditions to obtain them may easily be selected, yet the ratios being large or small do not necessarily prove that the materials are decidedly different.

In spite of the above seemingly adverse criticism of the paper, the writer wishes to repeat his opening statement that Mr. Borgeson is to be complimented for the development of this torsion test and this torsion testing machine.

MR. H. F. MOORE.[3]—I should like to call attention to certain stresses brought out in torsion tests, cantilever bending tests, and bending tests on beams with two symmetrical loads. In the cantilever bending test (or in a bending test of a beam with one central load) there are tensile and compressive stresses due to bending, and there are vertical and horizontal shearing stresses. In the beam with two symmetrical loads, between the loads the vertical and horizontal shearing stresses are zero, and there are diagonal shearing stresses. In the torsion test the stress commonly thought of is a shearing stress, which is a maximum at the surface of the twisted rod, but it is important to remember that on any diagonal plane there are also tensile or compressive stresses. For a circular bar in torsion the maximum tensile or compressive stress is at an angle of 45 deg. with the axis of the specimen, and is equal in magnitude to the maximum shearing stress set up by the twisting moment.

MR. P. H. BRACE.[4]—It seems to me that Mr. Borgeson has made a substantial contribution to the art of testing for purposes of control in factory operations.

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[3] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[4] Manager, Metallurgical Division, Reseach Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Repetitive manufacturing procedures are often more sensitive to variations in the quality of the materials being handled than are customary laboratory tests. Frequently, a variable which has escaped detection in the laboratory later crops up as a factor having a decided influence on manufacturing costs. Therefore, it is desirable that test methods of maximum sensitivity be available.

In Committee B-4 we are attempting to standardize a group of test methods and procedures for evaluating small differences in the properties of wire and strip. Three methods of applying bending have been studied. A torsion test may have some advantages over bend tests, but it does not lend itself to the detection of directional properties as of wire after having been coiled on a reel. In such wire there may be a decided difference between the stiffness in the plane of coiling and that in the perpendicular direction. The need for detecting such differences may in a given case determine whether the torsion test or the bend test should be used.

It would appear that in each type of test, complex stresses are involved. The choice of test method then would seem to hinge on the nature of the manufacturing operation to be served. Mr. Borgeson's torsion test comes into the field as a new possibility, supplementing the bend tests which Committee B-4 has been studying.

MR. S. E. BORGESON[5] (*author's closure by letter*).—The advantage of the torsion test is that it enables the resistance to distortion of a wire to be recorded as a single number in terms of a standard unit (pounds per square inch) independent of (1) the wire size, (2) the length of the specimen, (3) the testing machine used and (4) the direction of previous bends. It is recognized that, for certain purposes, a knowledge of the relative resistance to bending in various planes may be of interest, in which case a bend test must be used and the results given with reference to the position of the wire on the spool or coil on which it is wound. For most specification and control purposes, however, it was felt that an average value was preferable because of its simplicity.

Our remark, quoted by Mr. Tour, regarding the relation between the length of the specimen used in a bend test compared to its diameter was not intended as a criticism of the bend test but as a suggestion for its improvement. Changing the specimen length in the bend test in this way would enable the test results on various wire sizes to be made directly comparable.

Incidentally, the adjustment of the lever arm of the torsion tester proportional to the cube of the diameter of the wire is merely for the purpose of eliminating computation and is not essential. It is equivalent to the use of a slide rule for dividing by the cube of the diameter of the wire.

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[5] Mechanical Engineer, General Cable Research Laboratories, Rome, N. Y.

The percentage of maximum deformation at which to make torsion tests depends upon the use to which the wire is put. The value of 2 per cent was selected as the average maximum deformation to which the wire is subjected when used for purposes such as coil winding. It is also comparable to the average maximum deformation in a 15-deg. bend test.

It may be remarked, in connection with Mr. Moore's analysis of the stresses in bend and torsion test, that the use of a torsion test to indicate resistance to flexure is predicated on the assumption that work-hardening of metals is non-directional—that tensile strains, for example, increase the resistance to tensile, compressive and shear strains in the same ratio. While this is true for a perfectly annealed metal, it is no longer strictly true when work-hardening has proceeded far enough to produce preferred orientation. For this reason the results of torsion tests on hard wire will not necessarily bear the same relation to those of flexure tests, as on soft wire.

EFFECT OF TIME ON TENSILE PROPERTIES OF HARD-DRAWN COPPER WIRE

By Albert J. Phillips[1] and A. A. Smith, Jr.[1]

Synopsis

The object of the paper is to show the effect of time on the flow of hard-drawn copper wire under sustained loads. Also, an accurate determination of the modulus of elasticity has been made for both soft and hard-drawn copper wires. Test wires were fastened by swivel clamps at the top, threaded through brass tubing and dead weights suspended at the bottom by means of suitable clamps. Determination of strain was made with a portable microscope, measuring the distance between a knife edge at the bottom of the brass tubing and another knife edge on a small sleeve threaded over the wire. The gage length was 60 in. and the accuracy was ±0.000004 in. per inch.

From the data obtained, the following conclusions are drawn:

1. Hard-drawn copper wire will creep at room temperature for many days even under loads of only 50 per cent of the tensile strength.
2. The creep characteristics of different copper wires are similar for a given percentage of their respective tensile strengths.
3. Creep characteristics are similar for intermittently and continuously loaded wires for tensile loads not exceeding 78 per cent of the tensile strength.
4. The modulus of elasticity of certain hard-drawn copper wires is 18,000,000 lb. per sq. in. ±400,000 lb. per sq. in.
5. The modulus of elasticity of certain annealed copper wires is 13,400,000 lb. per sq. in. ±500,000 lb. per sq. in.

Introduction

Many uses to which copper is put are structural in character and consequently an accurate understanding of the effect of time on tensile properties is quite important. It is surprising to find the dearth of information with regard to flow of copper under sustained loads, particularly in the case of hard-drawn wire, which is commonly used in aerial transmission and distribution cable without reinforcement. In fact, there is even some question in the literature as to the correct modulus of elasticity for copper, for C. S. Smith states in the 1933 National Metals Handbook that "Young's modulus, according to Searle, is 17,600,000 lb. per sq. in. for hard-drawn copper, and 18,300,000 lb. per sq. in. for annealed copper. The International Critical Tables give an additional value, 13,940,000 lb. per sq. in. for hard-drawn copper, attributed to a private communication from the American Steel and Wire Co. This value is undoubtedly much more nearly correct than that of Searle, although further work is necessary to give a really reliable figure. The American Brass Co. uses the value 16,000,000 lb. per sq. in. as the modulus of elasticity for both soft and hard-drawn commercial copper wire." The General Cable Corp. in its bulletin "Copper Conductors for Rural Lines," states on page 52 that the modulus of elasticity of hard-drawn copper wire is 17,400,000 lb. per sq. in. Sayre,[2] states that the modulus of elasticity for hard-drawn copper wire is 19,100,000-6S lb. per sq. in. where S is the imposed stress.

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[1] Superintendent of Research, and Research Metallurgist, respectively, Research Dept., American Smelting and Refining Co., Maurer, N. J.

In the Research Laboratories of the American Smelting and Refining Co. an apparatus and technique have been developed for determining accurately the rate of flow of soft materials at room temperature over a long period of time. It was decided that this technique could be modified to afford a precise determination of both the elastic and plastic flow of other metals, particularly copper and its alloys. The method in brief consists of direct loading of the suspended test piece with dead weights and measuring initially the extension due to loading and, in addition, the progressive lengthening of the specimen under the sustained load over a considerable period of time.

MATERIALS

The results reported in this paper are on two sets of test specimens both of which were made from hard-drawn copper wire from ordinary electrolytic copper wire bars of the tough-pitch variety. Specific analyses were not made upon the samples but it is certain that the copper contains approximately 0.04 per cent oxygen and has a conductivity of 101 per cent in the annealed form. Both samples were drawn from rod hot-rolled to $\frac{1}{4}$ in. in diameter and the first wire drawn direct to finish gage (0.0285 in. in diameter). The second wire was drawn to 0.102 in. in diameter, annealed at 450 C. and then drawn to finish gage (0.0285 in. diameter). By this procedure wires of different tensile strength but of identical gage were obtained. The wires may have been, but were not necessarily, from the same heat.

APPARATUS

The apparatus used contained 20 test stations and had for its main structural member a 6-in. inverted channel iron supported horizontally 8 ft. from the floor by a rigidly braced steel I-beam column at each end and an intermediate brace in the center. A sketch of a single station is given in Fig. 1. Test wires were suspended from this channel on 7-in. centers by means of a short length of chain. The method of gripping the

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[2] M. F. Sayre, "Plastic Behavior in Light of Creep and Elastic Recovery Phenomenon," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, p. 559 (1933).

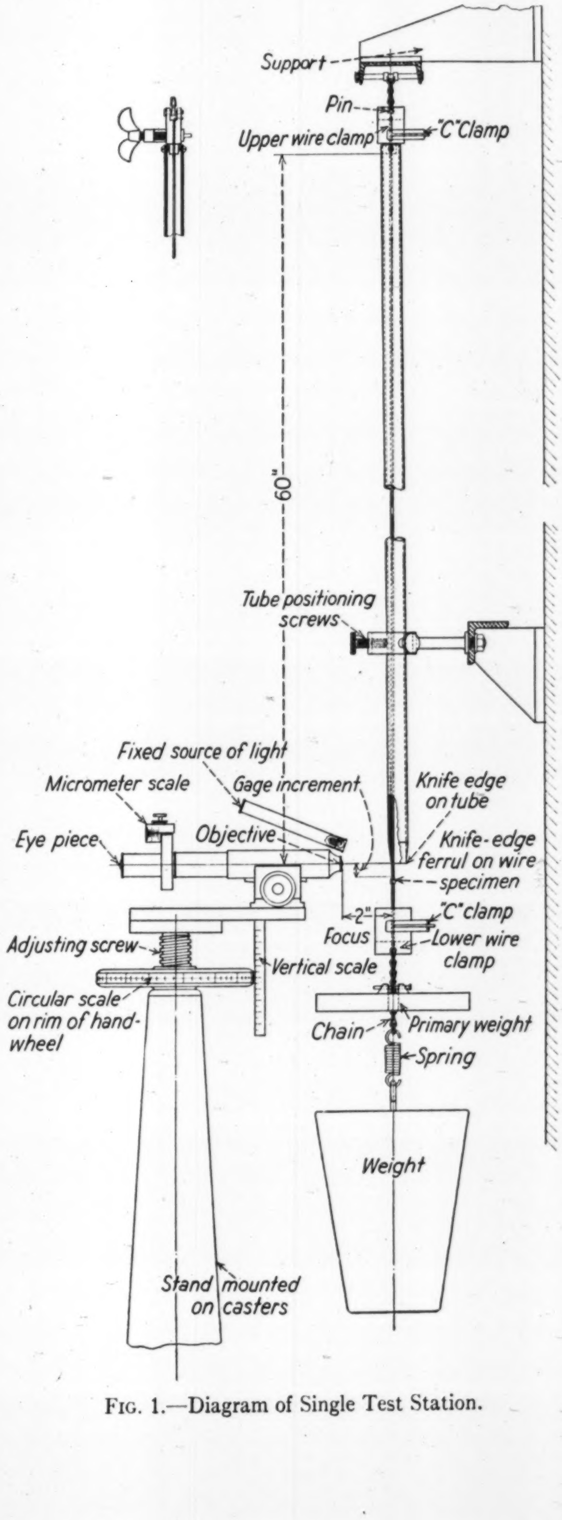
Support
Pin
Upper wire clamp
"C" Clamp
60"
Tube positioning screws
Fixed source of light
Micrometer scale
Gage increment
Knife edge on tube
Eye piece
Objective
Knife-edge ferrul on wire specimen
2"
Focus
"C" clamp
Lower wire clamp
Adjusting screw
Vertical scale
Circular scale on rim of hand-wheel
Chain
Primary weight
Spring
Weight
Stand mounted on casters

FIG. 1.—Diagram of Single Test Station.

wire was very important since it was imperative to avoid slipping, particularly in the upper grips, and at the same time avoid local damage to the wire in the grips which would result in premature failure. Satisfactory grips were finally prepared from suitable steel blocks by pressing between them a straight piece of piano wire of the correct diameter and employing between these grips, liners of fine emery paper. The lower $\frac{1}{2}$ in. of each pair of upper grips was turned down to a cylindrical nub $\frac{5}{16}$ in. in diameter concentric with the groove. The end of a 5-ft. length of $\frac{3}{8}$-in. brass tubing slipped over this nub and was suspended from it by means of two setscrews threaded through the walls of the nub of the grip. The lower end of the brass tube was turned to a knife edge and the tube was held lightly in a vertical position by means of three converging setscrews. The test wire was threaded through the tube and after sufficient load was applied to "plumb" the wire, the tube was adjusted by means of the setscrews so that its edge at the front just touched the wire. A small tubular sleeve likewise turned to a smooth precise edge was threaded over the lower end of the test wire and secured to it in close proximity to the large tube by a drop of lacquer.

The load was secured to the lower end of the test wire by grooved grips made of two pieces of flat iron held in place by a 2-in. C clamp and was suspended centrally by means of a short flexible chain. The load proper was divided into two parts. The smaller part was ring-shaped and was slipped over the chain and held in place by a cross pin. This weight plus the grips, clamp, chain and pins loaded the test wire to approximately 10 per cent of the tensile strength and served to establish an empirical and reproducible starting value for strain measurements. Experiments showed that this load applied overnight stretches the wire perfectly straight and does not cause detectable flow of hard-drawn copper after the first four or five hours. The major weight was suspended by a short spiral spring linked to the chain attached to the lower grips. This load was applied smoothly by means of a hydraulic jack.

Strain measurements were made by determining the distance between the lower edge of the large $\frac{3}{8}$-in. tube through which the test wire was threaded, and the upper edge of the tubular sleeve which was slipped over the wire from the bottom. As a general rule this marker was secured in place about 0.010 or 0.015 in. below the large tube after the minor (10 per cent) weight had been applied to the wire. The distance was measured accurately with a horizontal microscope mounted on a heavy portable cast-iron base riding on three ball casters. The microscope could be raised and lowered by means of a calibrated handwheel which could be read directly to 0.0025 in. and by interpolation to one-tenth of this amount. The microscope was equipped with a 42-mm. Leitz Micro Summar objective permitting a considerable depth of focus with a satisfactory working distance of about 2 in. The ocular was a Spencer screw micrometer eyepiece 25×. A tube length of 186 mm. was used in order to have each scale division within the ocular equal to 0.0025 in. (the same as the calibration of the handwheel used to raise and lower the microscope). There were thirty scale divisions within the ocular, each of which could be read directly to 0.01 by means of the screw micrometer giving a theoretical sensitivity of 0.000025 in. Actually the markers upon which the readings depended could not be sighted with such accuracy and it is conservatively estimated that the accuracy was 0.00025 in. As the gage length was 60 in. this gave an accuracy of 0.000004 in. per inch.

The room in which the tests were conducted was specially built for the purpose. Its location was in the basement of an office building in which there were no moving machines and absolutely no vibration of the specimens was detectable under what was considered to be the most extreme condition of shock in the adjacent surroundings. The floors and walls were of concrete and the only outside wall and the ceiling were thermally insulated with a special insulating board. There were no windows and the door was tightly fitted, of the refrigerator type. The room was heated electrically by six resistance elements placed on the floor. The location of these elements was determined experimentally to insure maximum uniformity of temperature throughout the room. Temperature control was by means of a Cenco De Khotinsky bi-metallic regulator operating a relay. It was found possible to control the temperature to $\pm\frac{1}{4}$ deg. Cent. However, the temperature varied with the elevation so that at the top of the test specimen it was $31\frac{1}{2}$ C. $\pm\frac{1}{4}$ deg. Cent. while at the bottom of the gage length it was 30 C. $\pm\frac{1}{4}$ deg. Cent.

DATA

In testing wire 0.0285 in. in diameter it is evident that an initial gage reading for zero stress is difficult, if not impossible, to obtain directly. Hence, the procedure of loading to approximately 10 per cent of the tensile strength before taking the initial strain reading was adopted as previously outlined. However, a careful study was made of the stress-strain relationship with loads down to 2 per cent of the tensile strength after preliminary stretching with loads of 50 per cent or more of the tensile strength and it was found that the wire was almost perfectly elastic in this range, permitting an accurate extrapolation to zero stress. All data given in this paper have been corrected to zero stress by this extrapolation.

In Fig. 2 the data obtained on the copper wire drawn hard from the hot-rolled rod are given in the form of strain-time curves with stress values properly shown. The tensile strength value is given for convenience on the zero time ordinate. The dotted portion of the curve marked 62,100 lb. per sq. in. indicates extrapolation, for the specimen broke during the night.

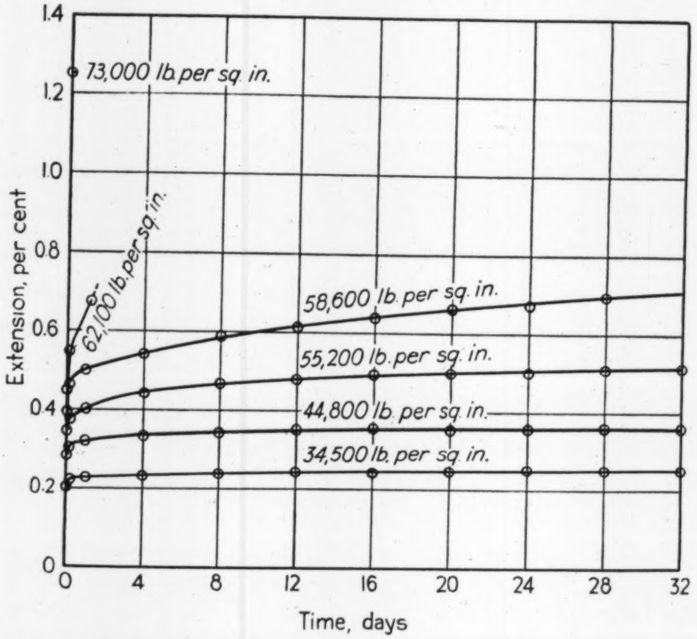

FIG. 2.—Plastic Flow of Hard-Drawn Copper Wire.

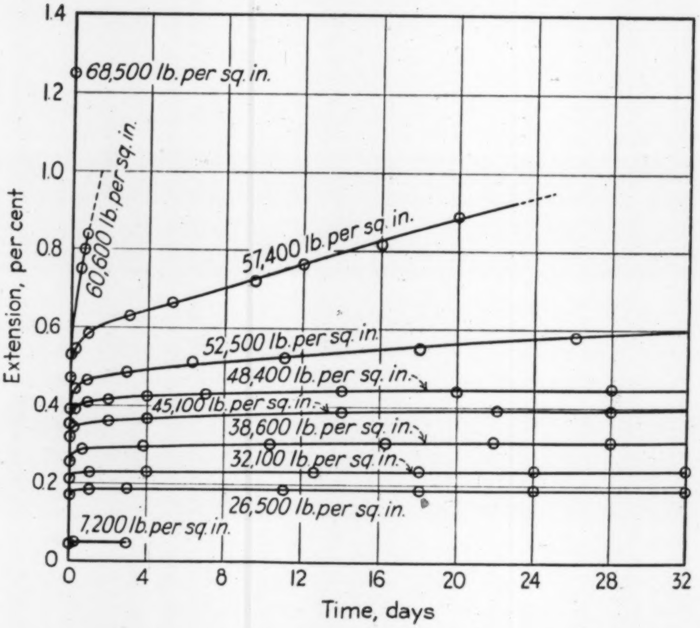

FIG. 3.—Plastic Flow of Hard-Drawn Copper Wire.

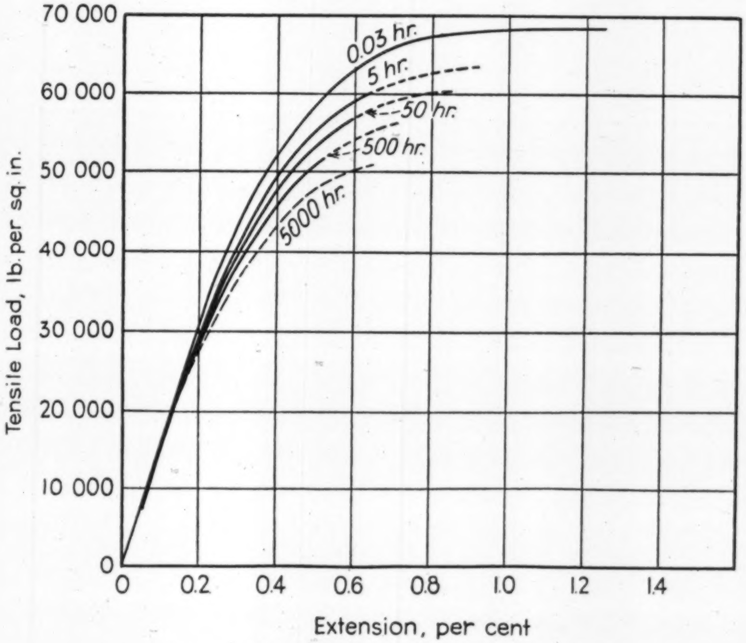

FIG. 4.—McVetty Diagram of Data of Fig. 3.

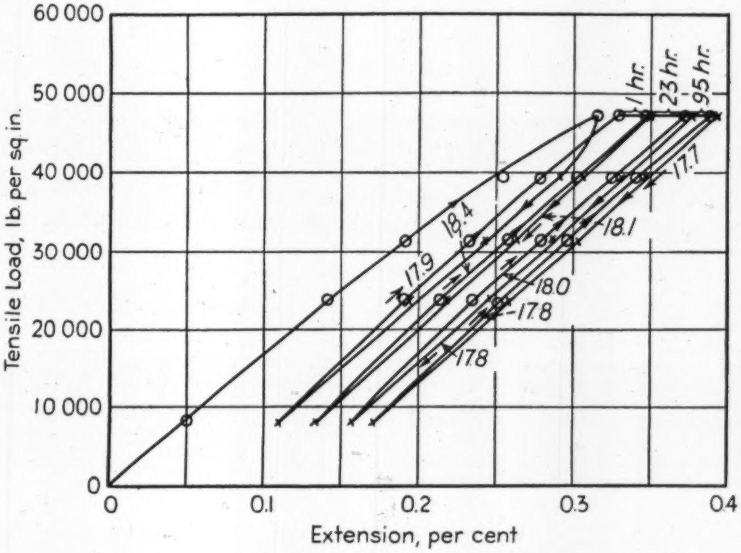

FIG. 5.—Elastic Properties of Hard-Drawn Copper Wire.

The numbers on the curves are the moduli of elasticity in millions of pounds per square inch.

It should be noted that the lowest curve, labelled 34,500 lb. per sq. in. does not show a zero rate of extension even after 32 days. The data prove, however, that the rate cannot be more than 0.0008 per cent per day and may be considerably less. The specimen loaded to 55,200 lb. per sq. in., approximately 75 per cent of its tensile strength, was extending at the rate of not more than 0.0013 per cent per day at the end of 32 days.

Figure 3 presents the data obtained from the copper wire annealed on 0.102 in. diameter. Many more strain readings than those shown were taken, particularly during the first few days of test. The sample loaded to 26,500 lb. per sq. in. was extending at a rate of not more than 0.00011 per cent per day after the second day.

From the data of Fig. 3, McVetty type stress-strain curves at various time intervals can be derived by interpolation and such curves are plotted in Fig. 4 and are represented by full lines. Dotted lines represent extrapolation which, although reasonably conservative, should be used with caution. An examination of this figure indicates the tremendous influence of time in determining the elastic and plastic properties of copper.

A separate study of these effects is illustrated by Fig. 5. In this experiment the copper wire that was hard drawn from 0.102 in. in diameter was preloaded to 8000 lb. per sq. in. and the initial reading taken after 13 hr. at this load. The stress was then increased in steps to the following values with strain readings after each increment: 23,700, 31,500, 39,300 and 47,200 lb. per sq. in. The specimen was then unloaded and reloaded as promptly as possible, taking the strain readings for each increment of stress, the complete cycle taking approximately 5 min. Stress-strain curves were then obtained after successive periods of 1, 23 and 95 hr. under the maximum load. The data are clearly represented in the figure and show that initially the rate of flow was large enough to influence greatly the shape of the stress-strain diagram. However, after 1 hr. at load, the rate of flow had become so small compared to the speed of the test that the wire appeared to be perfectly elastic. In fact, strain recovery was sufficiently rapid during the period of low stress to cause a slight but perceptible decrease in strain upon return to maximum load. This was confirmed by other experiments in which the maximum to minimum stress was obtained by a single load increment, thus permitting accurate gaging of the strain recovery at the minimum load. It was found that the recovery at minimum load was equal to or greater than the recovery at maximum load if a sufficient time interval were allowed.

Figure 5 indicates that there is no difficulty in determining accurately the modulus of elasticity for copper if the specimen is preloaded for a reasonable period of time. Several tests have shown that the instantaneous modulus of elasticity for hard-drawn copper wire at 30 C. is 18,000,000 lb. per sq. in. ±400,000 lb. per sq. in. Tests have also been

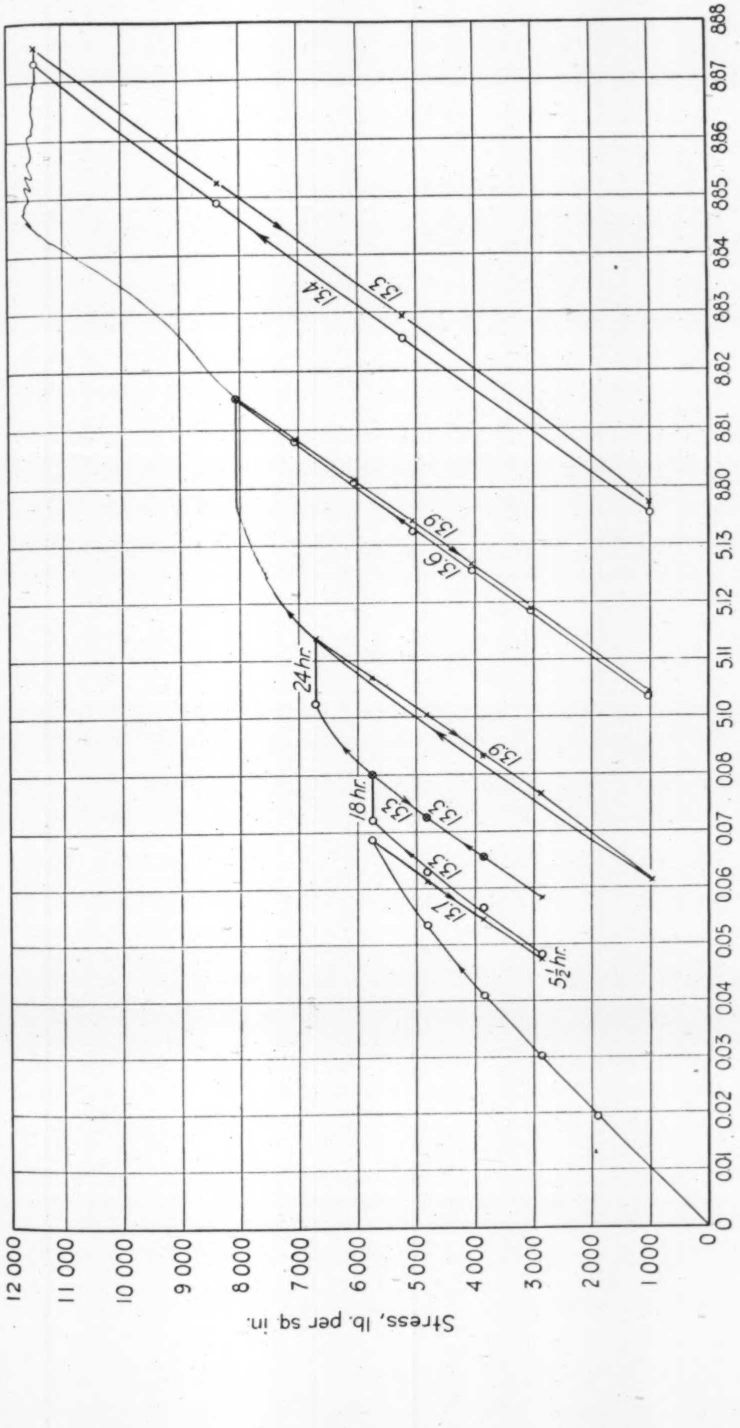

FIG. 6.—Elastic Properties of Annealed Copper Wire.

The numbers on the curves are the moduli of elasticity in millions of pounds per square inch.

made on copper wire annealed at 400 C. for 1 hr. and rapidly cooled in air, having a tensile strength of 32,000 lb. per sq. in. and the modulus has been determined as 13,500,000 lb. per sq. in. ±500,000 lb. per sq. in. The data are given in Fig. 6. Initially the stress used was only 25 per cent of the tensile strength of the soft wire and the total strain, both elastic and plastic, was less than 0.1 per cent. The wire was then loaded overnight to 50 per cent of its tensile strength, causing it to stretch 5 per cent. The modulus was then re-determined, loading to 33 per cent of the original tensile strength but calculating the modulus on the basis of the reduced area, obtaining a value of 13,400,000 lb. per sq. in. It was then re-loaded to 69 per cent of its tensile strength for 17 hr., causing it to stretch 8.7 per cent and the elastic properties again determined.

DISCUSSION

It was anticipated that from data such as is given in Figs. 2 and 3, it would be possible to predict a safe minimum period for which a given wire will support a specific load. Such a calculation would, of necessity, be based on a fundamental assumption that the extension to cause rupture is independent of time. However, the few tests to rupture completed to date have all shown extensions less than those obtained with the conventional tension tests and it is now felt that until more data have been accumulated it is not advisable to calculate stress-life curves. Such data will be released at a later date.

In addition to normal continuous load-time extension curves, engineers are interested in the effect of discontinuous and varying loads. For example, if a wire were loaded for periods of several days at alternately 10 and 70 per cent of its tensile strength, will the total extension be more, less or the same as would be obtained if the wire were loaded first at 10 and then at 70 per cent for a continuous period equal to the sum of the discontinuous periods? Several experiments were conducted to test this and the conclusion reached was that loads up to 78 per cent of the tensile strength will cause the same extension for the same total period of time at load. A certain degree of strain recovery is always experienced with time at low loads but upon reloading this recovery is exactly compensated by a short initial period of accelerated extension. However, with loads above 78 per cent of the tensile strength, intermittent application shows an accelerated extension for each re-loading cycle that more than offsets the strain recovery at low loads.

The experiments indicate that hard-drawn copper is instantaneously perfectly elastic. However, this fact is not apparent in a normal tension test due to the initially high rate of plastic flow even under moderate loads. The tremendous difference in modulus of elasticity between hard-drawn and annealed copper is surprising but it is felt that it is real and may even be fundamental. A few experiments have been conducted which indicate that different values will be obtained with different annealing technique. A study of this condition is now being made. In no case, however, have we encountered a modulus for annealed copper wire as high as that of the lowest modulus obtained on hard-drawn wire. This is surprising, particularly since preliminary experiments with brass and bronze wires, using the same technique, gives the usual higher value for the modulus of the annealed wire. It may be suggested that soft wire, lightly loaded, might show a false elasticity due to minute bends and kinks. However, it is unlikely that such effect would be of any considerable magnitude in wire stretched 5 per cent or more. Since the modulus of elasticity of copper wire depends upon the condition of the wire, this may account in part for the disagreements in the literature, but it is more probable that the differences reported were mainly due to failure of technicians to consider the time factor.

CONCLUSIONS

1. Hard-drawn copper wire will creep at room temperature for many days even under loads of only 50 per cent of the tensile strength.

2. The creep characteristics of different copper wires are similar for a given percentage of their respective tensile strengths.

3. Creep characteristics are similar for intermittently and continuously loaded wires for tensile loads not exceeding 78 per cent of the tensile strength.

4. The modulus of elasticity of certain hard-drawn copper wires is 18,000,000 lb. per sq. in. ±400,000 lb. per sq. in.

5. The modulus of elasticity of certain annealed copper wires is 13,400,000 lb. per sq. in. ±500,000 lb. per sq. in.

DISCUSSION

Mr. S. E. Borgeson.[1]—I should like to ask whether the authors have tried applying one of the formulas for the speed of testing to their results and whether the effect of loading the wire for a length of time is not similar to the effect of slow-speed testing in that the results are a function of the log of the ratio of the average speeds of testing.

Mr. D. K. Crampton.[2]—It is interesting to note the straightening effect of the initial application of a certain load on the stress-strain curve of hard-drawn wire. Presumably part at least of the effect results from the release or rearrangement of internal stress and it therefore would also be interesting to know whether tests on this same wire have been made following a very low temperature anneal commonly referred to as a relief anneal. I would expect that such a procedure also would show a straightening of the stress-strain curve and about to the same degree.

I should also like to ask whether, on the anneal at 100 C. where softening was observed, microscopic examination was made to determine whether or not there was visible recrystallization. It is surprising that measurable softening would occur at such a temperature which is well below any previously reported as being effective in this direction.

Mr. R. A. Schatzel[3] (*by letter*).—As pointed out by the authors, there is little published data on the flow of copper under sustained loads. We believe that the authors have made a worthwhile contribution and hope that the further data to be published will be presented at an early date.

On first reading the paper and finding the work had been conducted on wires of 0.0285-in. diameter, we were interested in checking the values against measurements made in the General Cable Laboratory, since for most uses wires of much larger diameter are used. We have made a number of determinations on solid wires of diameter 0.2294, 0.2043 and 0.1620 in. drawn to A.S.T.M. Standard Specifications for Hard-Drawn Copper Wire (B 1 - 27).[4] All were drawn from electrolytic copper of approximately 0.04 per cent oxygen content.

Wire Diameter, in.	Tensile Strength, lb. per sq. in.	Average Modulus of Elasticity, lb. per sq. in.
0.2294	61 000	17 300 000
0.2043	61 525	18 400 000
0.1620	62 000	18 007 000
0.2043[a]	54 000	17 600 000

[a] Medium hard drawn, conforming to Specifications B 2-27, 1933 Book of A.S.T.M. Standards, Part I, p. 768.

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[1] Mechanical Engineer, General Cable Research Laboratories, Rome, N. Y.

[2] Research Director, Chase Brass and Copper Co., Inc., Waterbury, Conn.

[3] Director of Research, General Research Laboratories, General Cable Corp., Rome, N. Y.

[4] 1933 Book of A.S.T.M. Standards, Part I, p. 763.

The above determinations were made on a specimen 14.5 ft. long in an Amsler hydraulic testing machine. A typical procedure was as follows:

Load increased to 50 per cent of breaking strength, held for 3 hr.
Load decreased to 5 per cent of breaking strength.
Load increased to 60 per cent of breaking strength, held for 3 hr.
Load decreased to 25 per cent of breaking strength, held for 1 hr.
Load increased to 65 per cent of breaking strength, held for 3 hr.
Load decreased to 5 per cent of breaking strength.
Load increased to 70 per cent of breaking strength.
Load decreased to 5 per cent of breaking strength.
Load increased to 80 per cent of breaking strength.

While the time under load has not approximated that used by Phillips and Smith, it is interesting that despite this and the fact that a much larger section is used the values of modulus as determined agree quite well.

MESSRS. ALBERT J. PHILLIPS[5] AND A. A. SMITH, JR.[5] (*authors' closure*).—We believe that Mr. Borgeson is referring to the formula on plasticity which states that the yield stress is a logarithmic function of the velocity of deformation. We did apply this formula by plotting breaking stress against the log of time to fracture, and found it to be only approximately correct. In fact, as more fractures are secured, the evidence of deviation becomes greater. This is not surprising as the formula refers to stress at a constant velocity of deformation whereas the tests given in the paper refer to constant stress at variable velocity of deformation.

In answer to Mr. Crampton, we did test the wire of higher tensile strength after annealing 1 hr. at 100 C. and found that this treatment did more than relieve internal stress as indicated by its decided decrease in tensile strength. However, no microscopic examination was made of the wire. The wire probably recrystallized to some extent and it acted similar to some of the fully annealed wires. Zeerleder and Bourgeois in their paper on "Effect of Temperatures Attained in Overhead Electric Transmission Cables,"[6] found that copper wire softens at temperatures as low as 80 C. More work is being done on the effect of annealing, using various kinds of copper wire.

We are pleased to note that Mr. Schatzel has confirmed our determination of the modulus of elasticity on hard-drawn copper wire, particularly since he used much larger diameter wires.

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[5] Superintendent of Research, and Research Metallurgist, respectively, Research Dept., American Smelting and Refining Co., Maurer, N. J.

[6] *Journal*, Inst. of Metals, Vol. 62, pp. 321–329 (1929).

IMPACT BEND TESTING OF WIRE

By W. J. Farmer[1] and D. A. S. Hale[1]

Synopsis

This paper comprises a discussion of a machine designed to make rapid determination of the ability of wire to resist permanent deformation by bending.

Two types of machine used in the industry for wire bend testing are described and their features discussed with regard to their suitability for use as standard test methods.

A bend tester operated by the impact of a pendulum has been developed by the Bell Telephone Laboratories in collaboration with Subcommittee IV on Mechanical Tests of the Society's Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Results of typical tests with this machine are given, together with information gathered from ultra-rapid motion pictures taken of the machine in operation.

It is concluded that the impact bending machine described offers a simple, rapid and accurate means of measuring the bending properties of wire and that the information acquired from the test is directly applicable to design problems.

Introduction

In a large majority of the applications in which wire is used, the bending characteristics of the material are of major significance. There has been an ever-increasing demand in recent years for a quickly operable test by which the essential properties of wire might be specified.

Subcommittee IV on Mechanical Tests of the Society's Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys was early interested in the problem. An investigation was undertaken with the intention of devising and specifying a standard form of test which could be used both by manufacturer and user and which would express the properties of the material in terms most pertinent to the applications in which the wire was to be used. The committee stipulated that the test must conform to a number of conditions: It must measure the property of the wire of most concern to the user; conditions must be reproducible; the test must be easily and quickly made and by unskilled operators if necessary. The actual device must be inexpensive, rugged (that is, long lived under factory conditions), easily portable, and self-contained.

At the request of the committee a study was made of the two testing

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[1] Bell Telephone Laboratories, Inc., New York City.

machines most generally used in the industry, the "bend tester" and the "resilience tester."

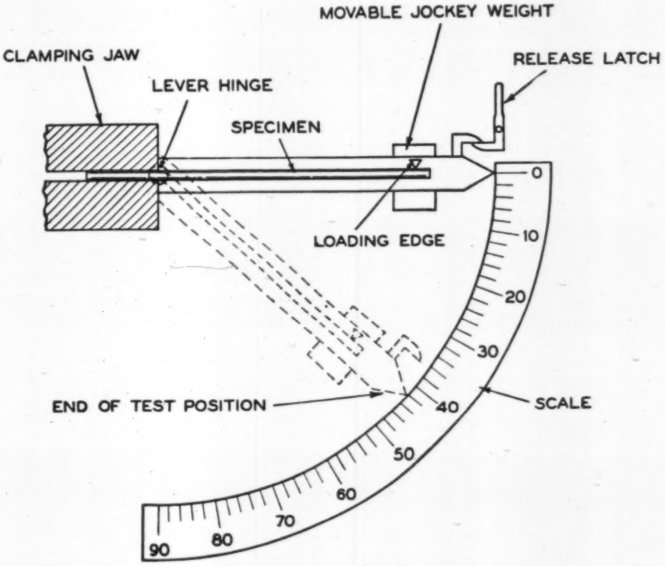
MOVABLE JOCKEY WEIGHT
CLAMPING JAW
RELEASE LATCH
LEVER HINGE
SPECIMEN
LOADING EDGE
0
10
20
30
40
50
60
70
80
90
END OF TEST POSITION
SCALE

FIG. 1.—Bend Tester Diagram.

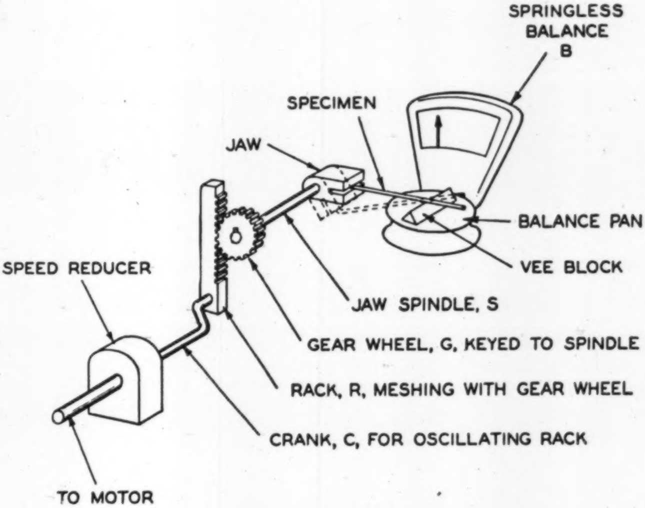
SPRINGLESS BALANCE B
SPECIMEN
JAW
BALANCE PAN
VEE BLOCK
SPEED REDUCER
JAW SPINDLE, S
GEAR WHEEL, G, KEYED TO SPINDLE
RACK, R, MESHING WITH GEAR WHEEL
CRANK, C, FOR OSCILLATING RACK
TO MOTOR

FIG. 2.—Resilience Tester Diagram.

PRELIMINARY INVESTIGATION

In the "bend tester," the specimen is held in a horizontal position by a clamp. A lever, hinged at the clamp face and carrying a horizontally projecting contact pin is caused to lean upon the specimen. The angle through which the specimen is bent by the weight of the lever is measured and taken as the bending index of the material. The essential features of this method are illustrated diagrammatically in Fig. 1. A model of this machine was tested and two operating difficulties were encountered. First, the end point or point of rest was hard to ascertain when specimens of small diameter were used because the specimen continued to bend under the weight for some time. With a 0.015-in. diameter copper wire the bend was not completed in 30 min. Second, the specimen was required to be just in contact with the loading edge, otherwise an impact blow was delivered instead of the steady load required.

The "resilience tester" indicates what appears to be the limit of elasticity in bending. Figure 2 is a diagrammatic representation of the apparatus. The specimen is clamped in the jaws secured to the spindle. This spindle is oscillated through a predetermined arc by the gear wheel, rack, crank, speed reducer and motor. A V-block is carried on the pan of a springless balance, its top edge being level with the axis of the spindle. As the spindle moves on its downward stroke the specimen is pressed against the V-block, causing the pointer of the balance to move. The movement is smooth to a certain point where there is a momentary hesitation, the indicator then moving on to the maximum which is determined by the angle of oscillation provided on the apparatus. The hesitation or "trip" point is taken as the measure of the "resilience" of the specimen. The angular movement of the specimen clamp must be carefully controlled or values for like materials are not reproducible on the same machine. This is understandable when it is considered that the specimen throws into motion the mechanism of the balance, the inertia of which is considerable. Although the angular velocity, length of specimen and total bend angle used in this machine were identical with those of the two others made some years previously, the balances were not of the same design. The difference in the balance mechanisms, because of their different inertias, apparently was responsible for disagreement in the results obtained on the machines. The balances used in the two older machines were of a model no longer obtainable. The standardization of such a test method would involve the standardization of the balances used, which would be a rather difficult problem.

Committee Recommendations:

With the results of the preliminary investigation available, the committee decided that a machine should be designed that would give a measure of the properties of the material in terms that could be applied directly in design. The machine was intended for testing materials to be used for vacuum tube and similar support wires which generally are subjected to bending strains only. The wires must be sufficiently stiff to withstand strains encountered in fabrication, shipping and in use in the field. Too much rigidity in the components, however, tends to transmit to the electrodes vibrations and thus to produce microphonic noise currents. The growing complexity of tubes calls for the maintenance of close and accurate spacing between elements. These are usually supported from the glass envelope by wires bent into the desired form by a tool or die having a definite and limited movement. The forming dies are so designed that a wire having a given thickness will spring back to the desired form upon release from the dies. These considerations make the control of the stiffness of the wire an obvious necessity; furthermore all the stresses encountered after fabrication are in the nature of impacts, not the impact due to an external body but to the inertia of the part when its motion is suddenly arrested. This type of stress is frequently encountered in tubes mounted in police car, airplane or other mobile radio equipment.

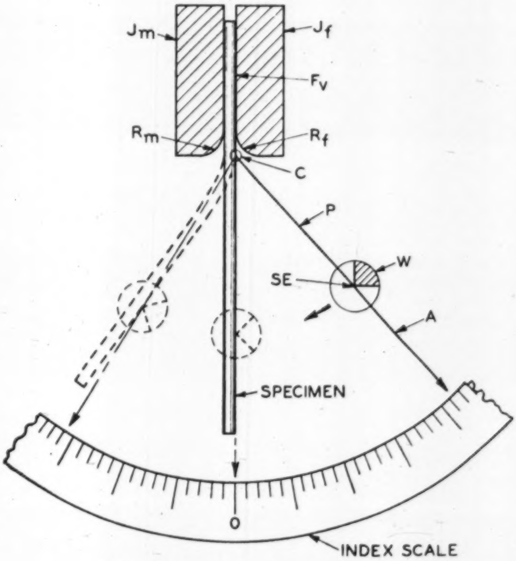

FIG. 3.—Bendometer Diagram.

In order, therefore, to simulate operating conditions an impact type of bend test was determined upon.

THE BENDOMETER

Figure 3 shows the essential elements of an impact bend testing device which has been given the name "Bendometer." This machine has been approved by Subcommittee IV of Committee B-4.[2]

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[2] See 1936 Report of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 157 (1936).

Referring to Fig. 3, the fixed and moveable jaws J_f and J_m, respectively, have vertical parallel clamping faces. The lower edges of both jaws are machined to 0.015-in. radius as shown at R_f and R_m. These radii are horizontally aligned. The value 0.015 in. was selected because sharp right-angled edges are almost impossible to realize. A radius was provided on both edges to prevent the prebending of the specimen during clamping. The jaws can be opened to 0.130 in. or closed entirely—they are parallel in all positions.

The pendulum system P comprises the arm A, which is made in the form of the letter U to permit the striking edge SE of the weight W to lie in the vertical plane of the fixed jaw face F_v when the pendulum hangs freely. The pendulum pivots are located at the open ends of the "U" and the pivot axis, C, lies in the intersection of the plane of the fixed jaw face F_v and the horizontal plane tangent to the corner radii R_f and R_m. This provides a fixed plane for the start of the bend irrespective of the diameter of the specimen. The pivots of the pendulum turn in self-aligning ball bearings in order to minimize friction. The pendulum arm is made of cold-rolled steel, case hardened, and indentations in the arm are provided for the weight locking screws. These locating indentations are $\frac{1}{2}$ cm. apart and, since the striking edge and weight are integral, they provide the means for adjusting the test length of the specimen, which is variable from 1 to 5 cm. in $\frac{1}{2}$-cm. intervals for the light weight and from $2\frac{1}{2}$ to 5 cm. in $\frac{1}{2}$-cm. intervals in the case of the heavy weight. A light pointer is provided on the pendulum which indicates the angular position of the arm at any time. When the pendulum hangs freely with no specimen in the machine the pointer indicates zero and is in the same vertical plane as the striking edge and the fixed jaw, F_v.

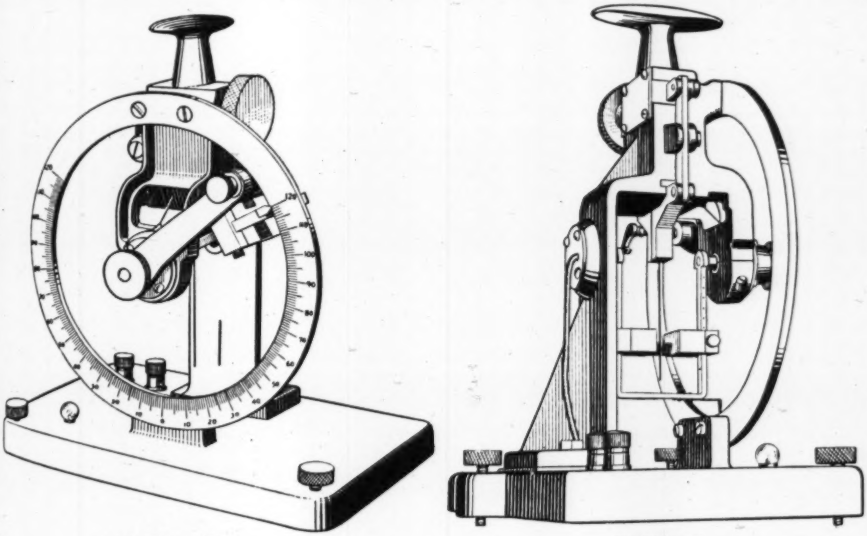
As mentioned above, each weight carries the striking edge as an integral part, the striking edge being located in the horizontal plane of the center of percussion of the mass. These weights, two of which are provided, one for light wires up to approximately 0.050 in. in diameter and one for 0.050 to 0.100 in. in diameter, are made of cold-rolled steel, case hardened. Two clamping screws are provided on each, which enter the indentations provided on the pendulum arms to lock the pendulum weight at the desired test length.

The machine is equipped with a graduated dial which permits the accurate determination of the height of fall of the pendulum and the amount of bend induced in the specimen. The dial scale zero is vertically in line with the pendulum axis and progresses upward to the right and left to 120 deg. in steps of 1 deg.

The latch used for holding and releasing the pendulum is mounted on an arm frictionally clamped to the boss carrying the front pendulum bearing housing. The latch is capable of being adjusted to release the pendulum from any angle between 0 and 120 deg.

To assist in aligning the specimen with the jaws, a crosswire is provided in an opening in the front bearing casting and a vertical white line is scored on the main support member. The specimen is aligned by sighting across the wire and the line.

When small wires are being tested a device must be provided for detecting the contact of the striking edge and the specimen. The operator's hand is not sufficiently sensitive to detect contact and the reading of bend may be several degrees too high (the specimen appearing too soft). To this end the pendulum system is electrically insulated from the body of the machine. A 2.2-v. lamp which is connected in series with the clamping jaws, striking edge and dry cells will light when contact is made between the specimen and the striking edge, the specimen forming the link closing the circuit. Two binding posts and the necessary connections provide for the use of a more sensitive contact detecting device, for example a pair of headphones or an electric relay.



(*a*) Front View. (*b*) Side View.

FIG. 4.—Bendometer.

Three screws are provided for leveling the instrument.

Figure 4 shows the Bendometer in its present form.

Physical Constants:

The combined weight of the pendulum and pointer is 32.4 g. and its center of gravity is located 6.72 cm. from the axis. The small weight is 112 g., the large weight 400 g., both weights include the locking screws. The center of gravity of both weights is located at the striking edge.

The energy available in the instrument was found by computing the center of gravity of the system and the vertical height through which it falls in the downward stroke. It may therefore be seen that the maximum potential energy of the instrument, when using the heavy weight, 5-cm. test length and a fall angle of 120 deg. is approximately 3300 cm-g.

Table I shows the location of the center of gravity of the system for the various test length for both small and large weights and the corresponding potential energy values.

TABLE I.—LOCATION OF CENTER OF GRAVITY OF SYSTEM AND POTENTIAL ENERGY VALUES.

Test Lengths, cm.	Distance of Center of Gravity of System from Axis, cm.		Potential Energy Values, cm-g.					
			120-deg. Fall Angle		90-deg. Fall Angle		60-deg. Fall Angle	
	Small Weight	Large Weight	Small Weight	Large Weight	Small Weight	Large Weight	Small Weight	Large Weight
1	2.28	....	495	....	330	....	165	....
1.5.....	2.67	....	579	....	386	....	193	....
2	3.06	....	663	....	442	....	221	....
2.5.....	3.45	2.81	747	1827	498	1218	249	609
3	3.83	3.28	831	2127	554	1418	277	709
3.5.....	4.22	3.74	915	2427	610	1618	305	809
4	4.61	4.20	1000	2727	666	1818	333	909
4.5.....	5.00	4.67	1083	3027	722	2018	361	1009
5	5.39	5.13	1167	3327	778	2218	389	1109

TABLE II.—MECHANICAL EFFICIENCY VALUES.

Weight, g.	Fall Angle, deg.	Efficiency, per cent				
		Test Length, 5 cm.	Test Length, 4 cm.	Test Length, 3 cm.	Test Length, 2 cm.	Test Length, 1 cm.
112........	120	97.3	96.8	96.8	96.8	95.8
	90	97.3	96.5	96.5	96.5	96.5
	60	97.0	97.0	97.0	95.7	95.7
	30	96.2	96.2	96.2	96.2	96.2
400........	120	97.8	97.8	97.3		
	90	98.2	98.2	98.2		
	60	98.5	97.0	97.0		
	30	96.2	96.2	96.2		

The efficiency of the machine is measured as the ratio

$$\frac{1 - \text{Cosine angle of rise of pendulum}}{1 - \text{Cosine angle of fall of pendulum}}$$

The ratio has been determined for a number of positions and the values are shown in Table II.

TEST PROCEDURE

In making tests, the procedure is as follows:

1. The desired pendulum weight, of 112 or 400 g. is selected and locked

in position on the pendulum arm to give the required free test length between jaw edge and striking edge.

2. The machine is leveled by means of the three leveling screws provided in the base. Transverse leveling is indicated by the spirit level attached to the base casting and the pendulum pointer should coincide with the dial zero when the leveling operation is complete.

3. The pendulum is latched at the required angle. This angle should give a fall sufficient to produce a permanent bend in the specimen of between 30 and 70 deg.

4. The specimen is then clamped in position. If the specimen is straight, no precautions need be observed other than to keep it within a degree or so of being vertical in the plane at right angles to the swing of the pendulum. When curved specimens are being tested the procedure is to rotate the specimen so that it is in line with the front sighting wire and the vertical sighting line on the rear standard, and then clamp tight. This aligning is automatically performed when wire is taken directly from spools, as the plane of the curve of the wire naturally assumes a position parallel to the clamping faces, the specimen then offers a straight projection when viewed from the front. The torsion produced in the specimen by this type of clamping has been found to produce no appreciable effect upon the bend angle produced. Theoretically the angle should be greater than for a straight specimen as the real length of the specimen is an arc instead of a straight line.

Should the necessity arise to test curved wire that cannot be oriented to contact the striking edge at the zero position, the specimen may be clamped and the pendulum carefully brought into contact by hand and the angular bias either added or subtracted from the final reading depending on whether the pendulum strikes the wire before or after reaching the zero point. In testing curved specimens care should be taken that the specimen touches only the striking edge during the bend.

5. The pendulum is released and allowed to strike one blow, the pendulum being caught on the rebound. The reading is obtained by carefully raising the pendulum until it just contacts the specimen.

INTERPRETATION OF RESULTS

Direct Reading of Bend Angle:

In accordance with the recommendation of Committee B-4, in shop practice and for standardization purposes, direct reference is made to the angle of bend of the specimen as measured in degrees. This makes for simplicity of operation and no calculation need be made. By this system the data on a specimen should be recorded thus:

Material Diameter, in. or cm.	Test Length, cm.	Weight, Large or Small	Fall Angle, deg.	Bend Angle, deg.

The recommendation that the bend angle be kept between 30 and 70 deg. was made in order that the most sensitive range of the instrument might be used, so that differences in stiffness will appear as real angular differences on the pendulum. The energy of the pendulum is equal to the vertical height of its center of gravity above the position it would assume when hanging freely, multiplied by the weight of the moving mass. At any point this value is given by

$$E = [R(1 - \cos\theta)]W$$

where E = potential energy,
R = radius of gyration of the system,
W = weight of pendulum system, and
θ = angle of pendulum measured from zero.

In a given test, with R and W fixed, the energy varies as $1 - \cos\theta$. The rate of change of this function when θ is 30 deg. is proportional to the angular change but it becomes increasingly disproportionate as θ approaches zero; as θ increases towards 90 deg. the function $1 - \cos\theta$ becomes increasingly disproportionate but at a much less rapid rate.

Geometric Stiffness Value:

The direct reading method set forth above is satisfactory for shop and general work, but the following is suggested for use where direct bend strength comparisons are needed at any point in the range of the instrument.

If the energy E is used in bending a sample to some particular angle, the value $\dfrac{E}{1 - \text{cos of angle of bend}}$ gives the average resistance offered by the sample. This reduces to dividing the vertical falling distance of the center of gravity of the system on its downward stroke by its rise when in contact with the specimen. Theoretically, then it follows that an unbendable sample will have a stiffness of infinity by this method as the angle of bend is zero. Conversely, a material offering no resistance whatever would have a stiffness of 1 because the vertical fall and rise angle and therefore the heights, would be equal.

Bend Test of Various Materials:

A number of materials were tested to determine the relative bending strengths. Wire 0.060 in. in diameter was used; the test length was 5 cm. and the fall angle was 120 deg. The large (400-g.) weight was used. Samples of each wire were tested before and after heat treatment. The heat treatment in all cases was a hydrogen anneal at 925 C. for 15 min. The results are shown graphically in Fig. 5.

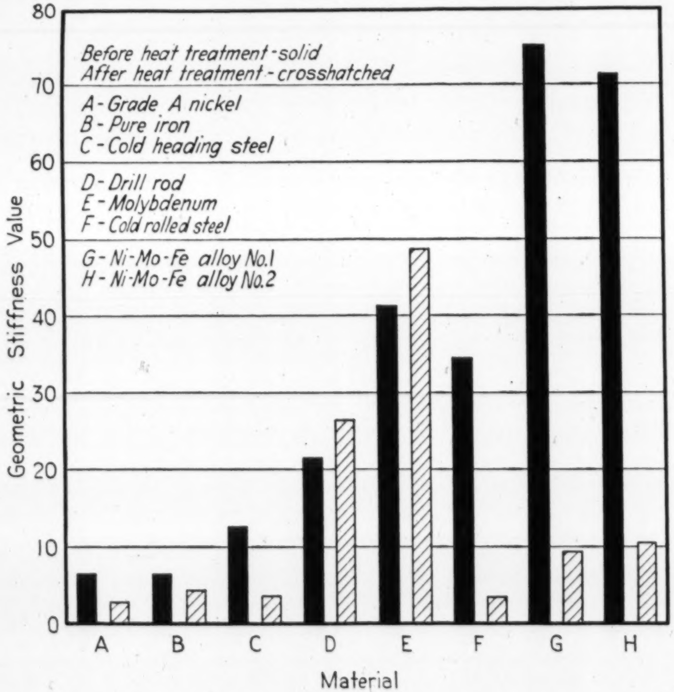


FIG. 5.—The Effect of Annealing on the Bending Strength of a Number of 0.060-in. Diameter Metal Wires.

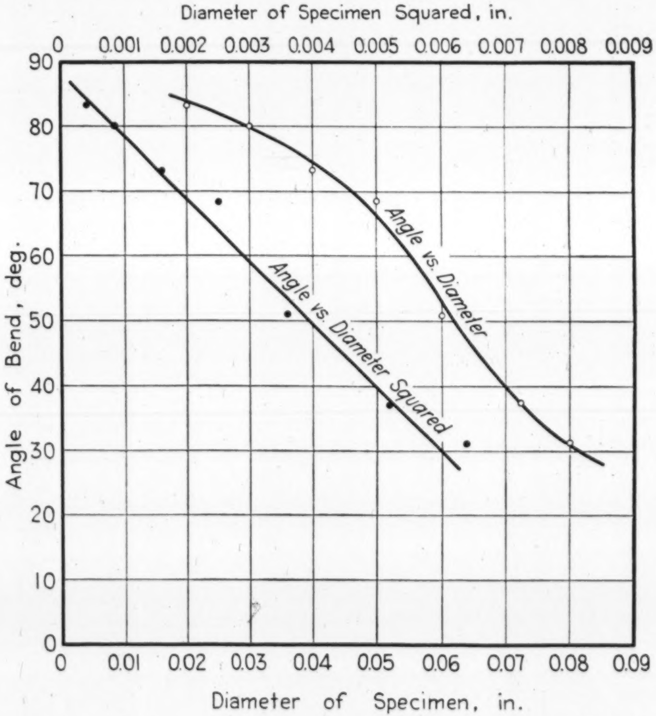
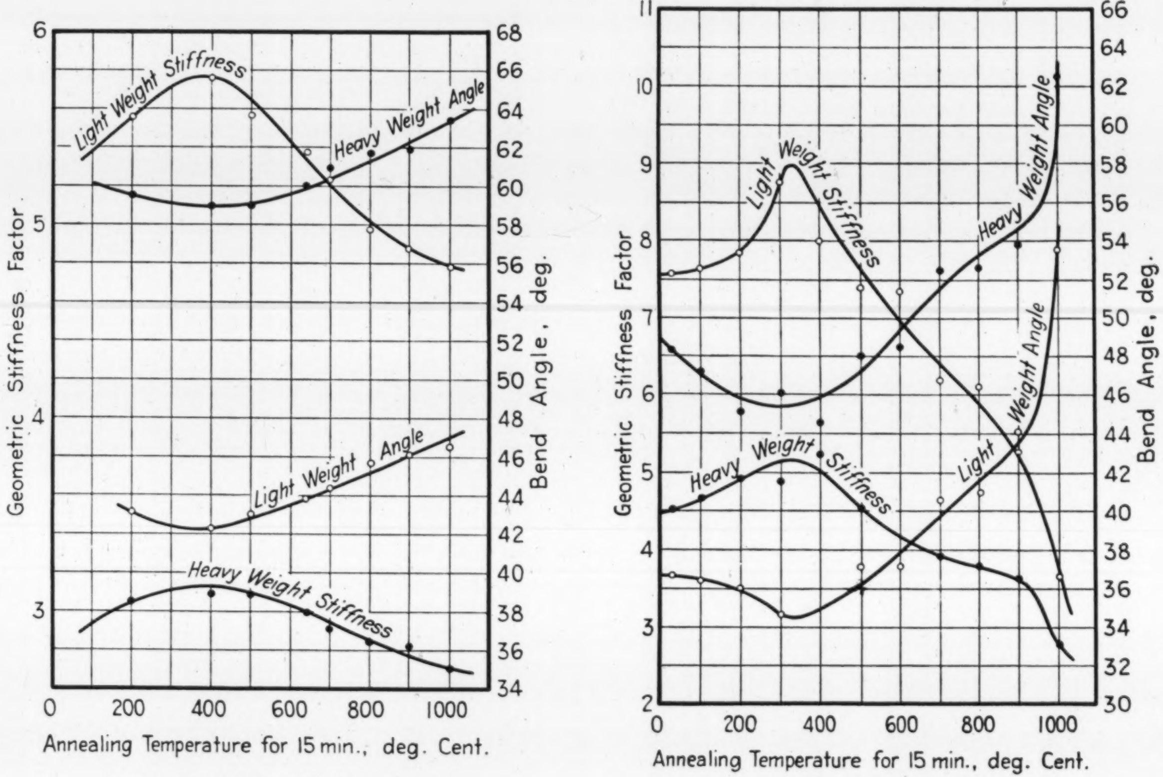


FIG. 6.—Effect of Diameter on Bending Strength of Grade A Nickel Wire.



(a) 2-per-cent stretch, hardened magno wire.

(b) 5-per-cent stretch, hardened magno wire.

Fig. 7.—Effect of Annealing on Wire.

Bending Strength as a Function of Diameter of Test Piece:

Samples of grade A nickel wire of various diameters were bent under identical conditions, test length 3 cm., fall angle 90 deg., 400-g. weight. The material was straightened on a rotary straightener, cut to length (3 in.) and annealed in hydrogen at 925 C. for 15 min. The data are plotted in Fig. 6.

Effect of Annealing on Bending Strength of a Nickel-Manganese Alloy Wire:

Magno wire 0.050 in. in diameter was used prepared as follows: Pieces 31½ in. long were cut from the spool and straightened in a rotary straightener. Each length was then cut into three 10.5-in. pieces and annealed in hydrogen at 925 C. for 15 min., care being taken to keep the pieces straight. Each 10.5-in. piece was elongated in a tension machine having a free length between jaws of 10 in. One lot was stretched to 10.2 in., and another to 10.5 in., giving 2 and 5 per cent increases in length, respectively. Specimens were divided into ten groups and annealed in hydrogen at 100, 200, 300, 400, 500, 600, 640, 700, 800, 900 and 1000 C., respectively, for 15 min.

They were then tested on the Bendometer under the following conditions:

Fall angle—120 deg.

Test No. 1—Large weight, test length 2½ cm.

Test No. 2—Small weight, test length 5 cm.

Four to six specimens were tested under each condition for both the 2-per-cent stretch and the 5-per-cent stretch lots. The results are given in Fig. 7.

To substantiate our findings further, ultra-rapid motion pictures were taken of the action of the machine while actually operating. The camera used was one in which a special chronometer dial is photographed coincidently with each subject view frame. The angular motion of the pendulum can thus be measured with relation to time. The speed of operation of the camera was 2000 frames per second and in projecting the pictures at the usual speed of 10 frames per second, a ratio of 1:200 is achieved. Qualitative examination of the pictures taken with no specimen in the jaws shows that the pendulum accelerates and decelerates smoothly. This is borne out by the efficiencies shown on Table II which indicate that a large proportion of the energy of the down-swing is retained in the up-swing. The energy losses are therefore due mainly to windage.

With the specimen mounted in the jaws, as under normal conditions, the pendulum was found to sweep against the specimen and bend it upward smoothly without chattering.

With the aid of the synchronized chronometer mentioned, the angular position of the pendulum *versus* the time was plotted for various combinations of free test length and weight. Smooth curves resulted for all conditions. The tests made with specimens in place showed a smooth transition from the falling curve to the bending curve, no abrupt change occurring.

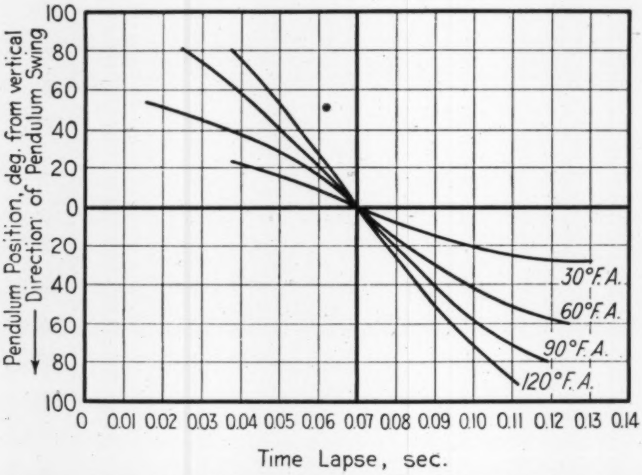


FIG. 8.—Time-Angle Relation, Light Weight, 5-cm. Test Length.

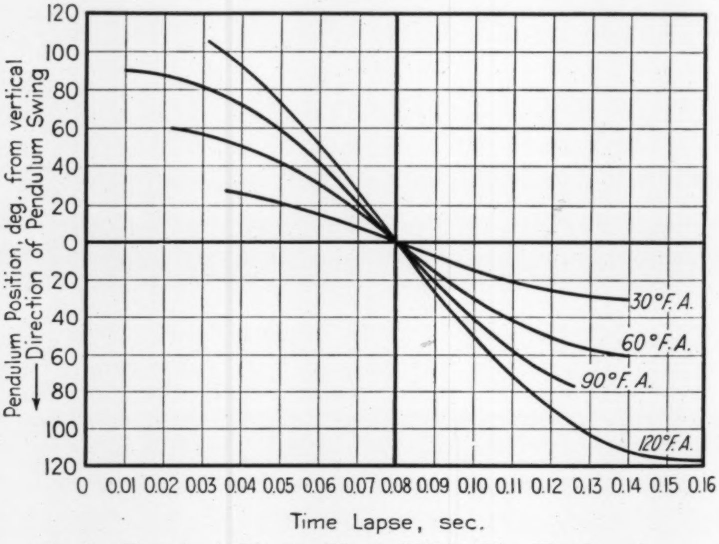


FIG. 9.—Time-Angle Relation, Heavy Weight, 5-cm. Test Length.

In all of the time-angle graphs the center abscissa represents the zero of the machine. The upper quadrant contains the downward stroke curves and the lower quadrant the upward or working stroke curves.

Figures 8 and 9 show curves of the time-angle relation of the machine empty; Fig. 10 gives the curves for actual working condition with the specimen in the jaws. The specimens were grade A nickel wire, 0.050 in. in diameter, 5-cm. test length in all cases.

CONCLUSIONS

The authors conclude that the pendulum-impact test described offers a valuable means of rapidly grading wire (and sheet strip) for use in apparatus where the bending properties are of importance.

The Bendometer fills the conditions outlined by Subcommittee IV of Committee B-4, and is based on sound mechanical principles.

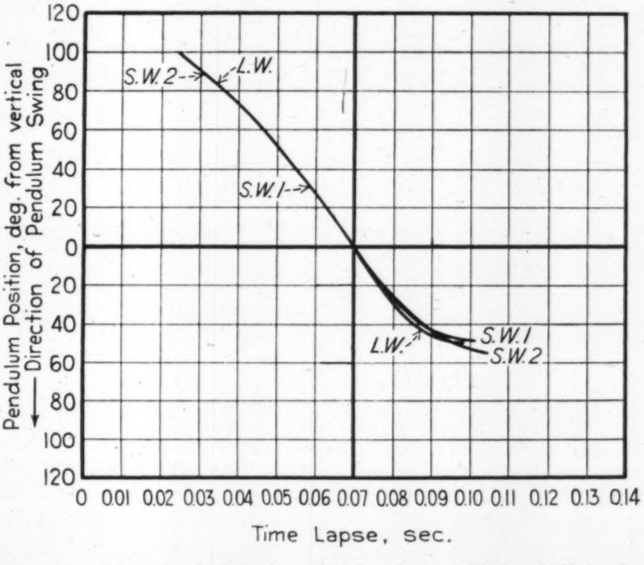


FIG. 10.—Time-Angle Relation with Specimen, 120-deg. Fall Angle.

A mass of data has been gathered during the last year or so while using the machine as a production control instrument, and fields of application other than the vacuum tube are rapidly opening up.

From the data given in this paper it appears that:

1. The efficiency of the instrument under no condition of operation falls below 95.7 per cent. From consideration of the smoothness of the velocity, Figs. 8, 9 and 10, and witnessing the projection of the ultra-rapid pictures, the loss due to mechanical vibration is seen to be small. The losses then are mainly friction and windage.

2. The response of different work-hardened materials to annealing varies widely according to the material itself. While under the conditions used, molybdenum became stiffer, nickel became softer and two nickel

molybdenum-iron alloys, although initially extremely stiff became quite soft. In selecting materials for support wires and the like, cognizance should be taken of the widely varying response to heat treatment.

3. Grade A nickel wire appears to vary in bending strength according to the square of the diameter of the piece. As the quality measured is plastic bend, which occurs beyond the elastic limit of the material, so uniform a curve as the one secured was hardly to be expected.

4. The amount of initial work hardening present in a piece of material greatly affects its response to annealing. This variation shows in the amount of change and, referring to the curves of Fig. 7 (*a*) and (*b*), the position at which the maximum stiffness appears. In Fig. 7 (*a*) the maximum stiffness is at 375 C., the work hardening being 2 per cent; in Fig. 7 (*b*) the maximum stiffness is at 325 C.—the hardening being 5 per cent. The indications are that the amount of work hardening should be considered in any application where annealing conditions are likely to be met.

DISCUSSION

Mr. S. E. Borgeson.[1]—I should like to ask how the authors took care of the fact that the energy used in testing two wires of different hardness, is different; that is, the energy used is the difference between the height of the fall and the height of recovery, so the stiffer of the two wires will be tested at the less energy?

Mr. D. A. S. Hale.[2]—The energy value at impact is a measure of the vertical fall of the pendulum system before contact with the specimen. After contact this energy is dissipated (neglecting windage and friction) in producing plastic and elastic bend in the specimen. We measure only the plastic bend.

Mr. M. A. Grossmann.[3]—The high-speed pictures showed that while the wire is bending it seems to move in jerks; I wonder whether that was due to the friction between the point of the pendulum and the moving wire, or whether there was any detectable periodicity in the deformation of the wire itself?

Mr. Hale.—The specimen presents a cylindrical surface to the impacting (bending) edge of the pendulum. Since the plane of the impacting edge is at right angles to the axis of the specimen, the area of contact is small. This results in a slight scraping action as the bend is made. The jerking is more noticeable in soft wires than in hard ones.

Mr. Sam Tour.[4]—A number of preliminary wire testing machines have been described by the authors of this paper. The appendix to the 1936 report of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys[5] gives three proposed methods for the bend testing of wire and describes a different machine used for each of the three methods. It might not be amiss at this time to call attention to the fact that all three types of test may be carried out on the one machine described under Method C. In addition to these types of tests, the machine described under Method C is also regularly used to develop complete stress-strain curves for the materials being tested. A universal machine of this kind, adapted to the carrying out of both static and dynamic tests, has many advantages.

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[2] Bell Telephone Laboratories, Inc., New York City.

[3] Director of Research, Carnegie-Illinois Steel Corp., Chicago, Ill.

[4] Vice-President, Lucius Pitkin, Inc., New York City.

[5] Proposed Method for the Bend Testing of Wire, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 162 (1936).

THE SPECTROGRAPHIC DETERMINATION OF MAGNESIUM IN ALUMINUM ALLOYS[1]

By W. R. Koch[2]

Synopsis

In order to compare the results of magnesium determinations in aluminum alloys by several spectrographic methods utilizing metallic electrodes, objective photometric measurements of the spectrograms were made photoelectrically. Suitable line pairs were selected for the determination of magnesium within ±10 per cent of the amount present over the range of concentration from 0.1 per cent to 4.0 per cent. The results obtained by the spark method were more accurate than those obtained by the arc method, although the arc method offered satisfactory accuracy within a more limited range of concentration. The presence of approximately 1 per cent of copper, iron, silicon, or manganese made no significant variation in the intensity relations of the aluminum and magnesium lines. In the several cases studied it was found that slightly greater accuracy resulted from the use of a pair of lines, both of which were spark lines, than from the use of a pair of lines, one of which was an arc line and the other a spark line.

Introduction

The use of emission spectra for the determination of the constituents of a luminous vapor is based on the characteristic relation of each spectrum line to a certain chemical element present in the source. Quantitative analytical methods have been developed on the basis of the intensities of the spectrum lines due to the chemical element being considered. Since reproducible excitation conditions in the source are essential, methods for the standardization of the source have been devised. The methods of photographic photometry developed in astrophysics and in atomic structure research have been applied in this field in order to furnish objective measurements of the photographic record. Although considerable progress has been made in the development of spectrographic methods of analysis it should be emphasized that spectrographic results are always based directly or indirectly on standards obtained by chemical analysis or synthesis. Because of this fact the standards used should be critically selected. For this same reason care must be exercised in the comparison of chemical results and spectrographic results in cases in which the standard samples are included in the comparison.

The spark method of spectrography has been applied to the analysis of aluminum alloys by several investigators. Triché[3] described a spectrographic method for the determination of magnesium in aluminum alloys based on a spark discharge between an antimony electrode and solutions of the aluminum alloys combined with various amounts of chromium solutions. The amount of chromium which it was found necessary to add to the solution of the sample in order to obtain equality of the intensities of a particular magnesium line and chromium line was used as a measure of the magnesium present. Data were presented by van Someren[4] on the determination of magnesium in aluminum alloys based on a spark discharge between metal electrodes. The percentages of magnesium necessary to produce equality in the line intensities of twelve pairs of aluminum and magnesium lines were tabulated. Baschulin[5] and his associates published results on the determination of magnesium in aluminum alloys by a method based on a spark discharge. An auxiliary coupling spectrum of the copper spark was used to supplement the aluminum spectrum. The amounts of magnesium which resulted in equality in the line intensities of three pairs of aluminum and magnesium lines and in five pairs of copper and magnesium lines were presented. Scheibe[6] described a visual method for the determination of magnesium in aluminum alloys. A polarization photometer was used for the spectrum line intensity comparison in the spark source. The results obtained by this rapid procedure were within ±0.1 per cent for concentrations of magnesium below 1.8 per cent.

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[1] Publication approved by the Chief of the Air Corps, U. S. Army.

[2] Junior Physicist, U. S. Army Air Corps, Wright Field, Dayton, Ohio.

The present paper includes a comparison of the results obtained for the determination of magnesium in aluminum alloys by spectrographic methods using a spark source and an arc source. The effect of the presence of certain other elements upon the spectrographic determination of magnesium has been studied.

MATERIALS

A series of thirty melts of aluminum alloys was prepared especially for this investigation. The aluminum used was high-purity material containing only 0.03 per cent of other elements. It was obtained from the Aluminum Company of America. Each melt consisted of approximately 700 g. The constituents were melted in a graphite crucible in an electric furnace. Small vertical ingots 5 cm. (2 in.) square were poured from each of the melts. Chemical analyses indicated that the ingots were homogeneous. The chemical analysis of the alloys was carried out by the Chemical Unit at Wright Field by means of the pyrophosphate method. Electrodes approximately 1 cm. (0.4 in.) square were cut from the ingots for the spectrographic procedure.

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[3] H. Triché, "Analyse spectrale quantitative de métaux en faible proportion dans les alliages légers: nouvelle méthode de dosage, détermination du magnésium dans l'aluminium et le duralumin," *Bulletin* de la Société Chimique de France, Series 5, Vol. 1, pp. 495–505 (1934).

[4] E. H. S. van Someren, "The Spectrographic Analysis of Some Alloys of Aluminium," *Journal*, Inst. Metals, Vol. 55, pp. 265–274 (1934).

[5] P. Baschulin, A. Baskakow and A. Striganow, "Die Methode der quantitativen Spektralanalyse als Mittel zur raschen Bestimmung der Beimengungen von Mg, Mn, Si und Fe in Duraluminium," *Technical Physics* of the U. S. S. R., Vol. 1, pp. 108–117 (1934) (Published by the State Technical Theoretical Press, Leningrad).

[6] G. Scheibe and A. Schöntag, "Quantitative chemische Spektralanalyse von Aluminiumlegierungen," *Metallwirtschaft*, Vol. 15, pp. 139–141 (1936).

The series of alloys included seventeen melts of aluminum and magnesium distributed throughout the range of 5 to 0.01 per cent magnesium. The remainder of the series consisted of certain of the above melts with additions of approximately 1 per cent of elements frequently present in aluminum alloys. The elements studied included copper, iron, silicon, and manganese. The compositions of the alloys are given in Table I.

Table I.—Chemical Composition of Aluminum Alloys.

Alloy	Magnesium, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent
X106	....	0.01	0.01	0.01	....
X107	5.42	0.01	0.01	0.01	....
X108	4.95	0.01	0.01	0.01	....
X109	4.44	0.01	0.01	0.01	....
X110	3.97	0.01	0.01	0.01	....
X111	3.48	0.01	0.01	0.01	....
X112	2.99	0.01	0.01	0.01	....
X113	2.52	0.01	0.01	0.01	....
X114	2.00	0.01	0.01	0.01	....
X115	1.51	0.01	0.01	0.01	....
X116	1.28	0.01	0.01	0.01	....
X117	1.00	0.01	0.01	0.01	....
X118	0.77	0.01	0.01	0.01	....
X119	0.48	0.01	0.01	0.01	....
X120	0.26	0.01	0.01	0.01	....
X121	0.10	0.01	0.01	0.01	....
X122	0.035	0.01	0.01	0.01	....
X123	0.007	0.01	0.01	0.01	....
X124	4.11	0.01	0.01	0.01	0.43
X125	1.59	0.01	0.01	0.01	0.29
X126	0.22	0.01	0.01	0.01	0.15
X127	3.98	0.01	0.97	0.01	....
X128	1.37	0.01	0.86	0.01	....
X129	0.27	0.01	0.88	0.01	....
X130	4.55	1.06	0.01	0.01	....
X131	1.34	0.90	0.01	0.01	....
X132	0.26	1.05	0.01	0.01	....
X133	4.15	0.01	0.01	0.90	....
X134	1.45	0.01	0.01	0.88	....
X135	0.23	0.01	0.01	0.82	....

Procedure

The set of electrodes of each alloy which was used for the spark method was prepared according to the suggestion of H. V. Churchill of the Aluminum Research Laboratories. One surface of each electrode was polished on a metallographic sander in order to facilitate maintenance of the discharge at one point. The upper electrodes were machined from high-purity aluminum in the form suggested by A. W. Petrey of the Aluminum Research Laboratories. Rods of $\frac{1}{4}$-in. diameter were machined for a $\frac{1}{2}$-in. length to a wire-like projection of about $\frac{1}{16}$-in. diameter. The end of the projection was polished in a plane normal to the axis of the electrode.

The spark equipment used consisted of an auto-transformer (General Radio Co. Variac Type 200C) and transformer (1:112) operated from the 110-v., 60-cycle, alternating-current supply. The input of the transformer during the discharge was a current of 3.7 amp. at a potential of 130 v. A condenser of 0.02μf capacity was connected in parallel with the spark. The 2-mm. spark gap was maintained between the two electrodes in all cases. Exposures of 10 sec. were made.

The arc electrodes were not machined but were merely sawed from the ingots. The discharge between two similar electrodes was compared with that between one electrode of the sample and an upper electrode of high-purity aluminum. Other experiments with the upper electrode of graphite or high-purity carbon indicated that the use of such a material resulted in a more stable discharge from the standpoint of oxide formation. Since no significant advantage was discerned in the use of the high-purity carbon, the upper electrodes used for the arc spectra were $\frac{5}{16}$-in. diameter regraphitized graphite rods. The arc discharge carried 2.5 amp. with a drop of 70 v. across the gap which was supplied by the 220-v. direct-current supply. The nature of the discharge was satisfactory for maintenance of the arc during the 30-sec. exposures.

At the suggestion of J. B. Johnson, Chief of the Materials Branch at Wright Field, the gas sheath utilized in certain welding technic was considered as a protection against oxide formation and the resultant difficulties. Preliminary experiments on an arc in nitrogen indicate that the discharge is steadier and may be more subject to control than the usual arc. The spectra produced by this source were distinguished by an unusual freedom from background due to faint bands. However, the initial density measurements on this series of alloys were more scattered than those of the corresponding discharge in air.

The quartz spectrograph used in this investigation was the 1.5-m. focal length Littrow (E384 Hilger) instrument. The range of spectrum included on the 10-in. plate was that of 2500 to 3400 Å. The Wratten "M" photographic plates used were developed with metol-hydroquinone developer (D-19) to a contrast of approximately unity gamma. The slit width used was about 0.03 mm.

The photometric equipment used was the photoelectric recording microphotometer by Zeiss. The linear magnification of the record was 10 while the ratio of full excursion to the width of the filar image was 2800. The photometered area was 0.80 by 0.02 mm. The dark cell and clear plate deflections were exposed twice on each photometer record and densities calculated on the basis of these deflections. All measurements were made relative to the dark cell position without any attempt at correction for background. Density determinations were usually reproducible to within 0.01. All references to density in this paper refer to the logarithm of the opacity, which is the reciprocal of the transmission.

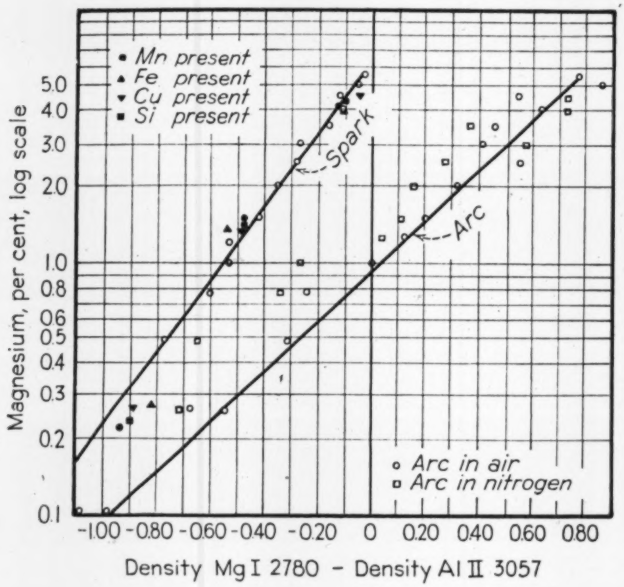

FIG. 1.—Relation Between Percentage of Magnesium and Density Difference of Pair of Lines in Arc and Spark Spectra of Aluminum Alloys.

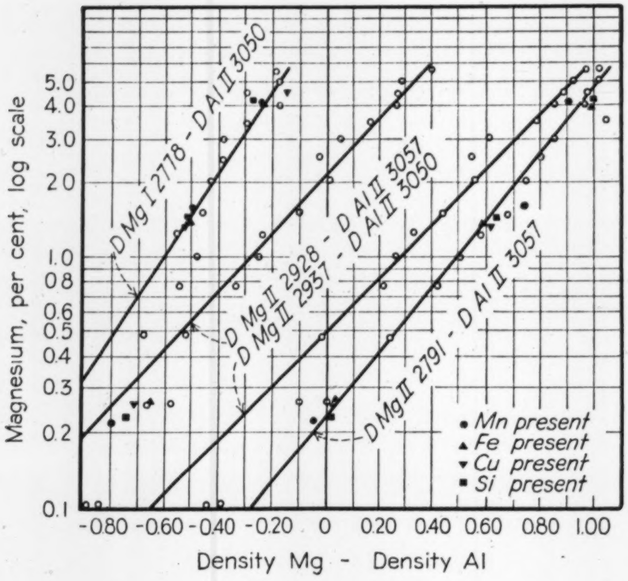

FIG. 2.—Relation Between Percentage of Magnesium and Density Difference of Various Pairs of Lines in Spark Spectra of Aluminum Alloys.

DATA AND DISCUSSION

The data obtained included the densities of twelve selected aluminum lines and twelve selected magnesium lines for the arc and spark spectra of the alloys studied. The data presented in Figs. 1 and 2 were selected as typical of the many combinations possible from the data available.

Since the determination of percentage composition from spectral data necessitates interpolation, it was considered desirable to plot the data in such a form that a linear relation would result. The basis selected was a semi-logarithmic graph of the relation between the logarithm of the percentage of magnesium and the difference in photographic densities of two selected spectrum lines, one due to magnesium and the other due to aluminum. Each point given in Figs. 1 and 2 represents a pair of single density determinations from a single spectrum of the alloy concerned. In routine work a considerable improvement in the precision of results can be obtained by using the mean of the values from two spectra or preferably the median of the values from three spectra.

The relation between percentage of magnesium and the density difference of a particular line pair is shown in Fig. 1. With a single exception data for the spark spectra fall within the ±10 per cent variation zone above a magnesium content of 0.5 per cent. The variation below this value is probably due to the lower photographic densities involved which fall on the curved portion of the characteristic curve of the photographic plate. It should be noted that the data for the alloys containing copper, iron, manganese, or silicon are consistent with those for the other alloys. The data for the same pair of lines in the arc spectra of the alloys exhibit more variation, with a number of points being located outside of the ±10 per cent zone. However, at low percentages arc data do not show the variation caused by the low density effect, partially because of the increased exposure time. The data for the same pair of lines in the arc spectra produced in nitrogen are included although the results are widely scattered. The pair of lines used in this comparison of the three discharges is composed of one line due to the magnesium atom and another line due to the aluminum ion. This choice was not made as representing the procedure for optimum analytical results, but in order to provide a line pair whose relation was not radically different in the various discharges.

In order to determine the effect of the choice of line pairs on the analytical results, the data in Fig. 2 were plotted for four line pairs from spark spectra. One pair containing a magnesium arc line and an aluminum spark line was chosen while the other three pairs represent spark lines exclusively. Although the variation is not outstanding it appears that somewhat better results are obtained when similar lines are used for comparison. In this case the low density effect is probably partially responsible for the variation since this line pair includes the least intense line considered. It should again be noted that the data for the alloys containing copper, iron, manganese, or silicon are consistent with those for the other alloys. The result obtained for equality of the line pair Mg II 2937—Al II 3050 shown in Fig. 2 is 0.50 per cent. This value compares favorably with the result of 0.50 per cent obtained by van Someren[4] and that of 0.55 per cent obtained by Baschulin.[5]

CONCLUSIONS

The effect of the presence of approximately 1 per cent of copper, iron, manganese, or silicon on the spectrographic determination of magnesium in aluminum alloys is negligible.

Comparison of the results obtained from spectrum line pairs composed of two spark lines with results obtained from combinations of arc and spark lines indicates that pairs consisting of like lines are more suitable for analytical purposes. However, there are frequently other factors of greater significance in the selection of line pairs.

Although the results obtained in this investigation do not indicate that the methods described, without further control of the source, are as accurate as conventional chemical methods for appreciable amounts of magnesium, the spark method is satisfactory for routine analysis where an accuracy of ±10 per cent of the amount present is sufficient. Except in the case of extremely low densities, the results are reliable within the range of concentration from 0.1 per cent to 4.0 per cent. In all cases the spectrographic method results in a considerable saving of time.

The arc method is restricted to more limited ranges of concentration for a given line pair. The principal advantage of the arc method is its greater sensitivity.

Acknowledgment:

The author takes pleasure in acknowledging the aid of J. B. Johnson, Chief of the Materials Branch at Wright Field, under whose supervision the investigation was conducted, and the cooperation of the members of the staff at Wright Field, particularly H. H. Stang of the Chemical Unit.

DISCUSSION

Mr. F. H. Emery.[1]—I should like to ask Mr. Koch for information concerning plate calibration and microphotometer technique.

Mr. W. R. Koch.[2]—The density of the plate is the common logarithm of the opacity. The opacity is the ratio of the incident light intensity to the transmitted light intensity as measured by the microphotometer. The dark cell position of the electrometer thread is the index from which all measurements are made. This position represents the condition of no light passing through the photographic plate. The greatest deflection from this position is that corresponding to the clear area adjacent to the spectrum. If we denote this distance by a and the deflection from the dark cell index to the peak (or dip) for a given line by b, the values of the transmission and the opacity are given by the ratios b/a and a/b, respectively. The substitution of deflections for light intensities is valid since the relation between them was found to be linear for the photometer used. The density is calculated as the common logarithm of the opacity. The difference of the densities determined in this manner for each line is used in the comparison of two spectrum lines. Such differences are plotted as abscissas in the figures.

Determination of the density difference as outlined does not require calibration of the plate. It does not furnish information as to whether the points involved are located on the straight-line portion of the plate characteristic. Independently it was found that all of the line pairs reported were located on the linear portion of the photographic curve with the exception of those containing the low density lines mentioned previously. This was accomplished by actually photometrically calibrating each individual plate.

Mr. A. W. Petrey[3] (*presented in written form*).—I am very much interested in Mr. Koch's statement that in evaluating the photometer records, all measurements are made with respect to the dark cell position. I should like to ask whether the records used in this investigation indicated uniform background densities. In work done in the Aluminum Research Laboratories along lines similar to Mr. Koch's investigation, we have preferred in many cases to measure from the background near the spectrum line, using the peak deflection from this point as a measure of the density of the line. Background densities must naturally be very uniform to use this method and the details of exposure have been studied very carefully to improve the precision of reproducing the background density.

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[1] Spectrographer, National Smelting Co., Cleveland, Ohio.
[2] Junior Physicist, U. S. Army Air Corps, Wright Field, Dayton, Ohio.
[3] Spectrographer, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

MR. KOCH.—The background densities were not considered in the calculation of the densities reported. The plates of this investigation had background densities which were quite uniform although they were not identical. Photometric work carried out in our laboratory indicated that it was extremely difficult to determine background corrections satisfactorily. Such difficulties are probably due to the fact that the background densities are located on the extreme low density end of the characteristic curve of the plate. In an effort to insure that the entire photometric measurement be objective and that no arbitrary or personal selection of a background density corresponding to a particular line enters into the result we have used the method as outlined. Since the density difference of a pair of lines is the significant quantity in analytical work, the absolute level of the background is not as important as the relation of the backgrounds corresponding to the lines which compose the line pair.

DEVELOPMENTS IN THE QUANTITATIVE ANALYSIS OF SOLUTIONS BY SPECTROSCOPIC MEANS

By O. S. Duffendack[1] and K. B. Thomson[1]

Synopsis

Two spectroscopic sources for the quantitative analysis of solutions are described. One consists of a pair of jets from which the solution is made to flow at a uniform rate from reservoirs moved by means of a synchronous motor. A highly inductive spark between the jets is photographed by means of a suitable spectrograph and the analyses are based upon the relative intensities of selected spectral lines of the test elements and of internal standards. Typical results from the use of such a source are given and the distribution of errors in the determinations of two elements is shown graphically.

The second source consists of a high-voltage alternating current arc between very pure carbon electrodes upon which a drop of the solution under analysis has been evaporated. This source, which is not capable of quite so high a degree of accuracy as the former, has the advantage in that only a drop of solution is required for analysis. Two other sources that require only a drop of solution for analysis were tested and are briefly described.

Introduction

Spectroscopic methods are finding ever increasing application and use. With this increase in application comes a demand that spectroscopic methods shall be as reliable and as precise as chemical methods of analysis. The methods and instruments of photographic spectrophotometry have been developed to the degree that sufficient accuracy in the measurement of the relative intensities of spectral lines can now be attained to permit the demands of precision to be met provided the spectroscopic source employed is sufficiently constant. Therefore considerable attention is being given to the development of suitable spectroscopic sources and this paper describes several sources that have been developed especially for the analysis of solutions.

The method of analysis employed is one using an internal standard.[2] The relative intensities of selected lines of the elements under analysis and of the internal standards are measured and their ratios applied to a working curve which expresses the relation between these ratios and the abundance of the elements in solution. The relative intensities were measured with the aid of a stepped slit as described by Thomson and Duffendack[3] and the general method of analysis followed is that of Duffendack, Wolfe, and Smith.[4]

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[1] Associate Professor of Physics, and Research Physicist, respectively, University of Michigan, Ann Arbor, Mich.

[2] W. Gerlach, "Zur Frage der richtigen Ausfuhrung und Deutung der quantitative Spektralanalyse," *Zeitschrift für anorganische und allgemeine Chemie,* Vol. 142, p. 383 (1925).

SPARK BETWEEN TWO JETS

In a recent paper, Duffendack, Wiley, and Owens[5] described a spectrographic method of analysis for biological solutions in which a spark source adapted from that described by Twyman and Hitchen[6] was employed. A highly inductive spark was struck between a metal electrode (silver) and an electrode consisting of the solution to be analyzed, and these authors point out the necessity of the careful cleaning of the metal electrode and of the shielding out of the light from that part of the spark close to the metal electrode. The spectral lines of the metal appear in the spectrum, and, because of the condensation of salts from the solution being analyzed on the metal electrode during the exposure, the spectral lines of the elements under test varied in intensity during the time of exposure in the light from the region near the metal electrode. Hence precaution was necessary to exclude the light from this region from the spectrograph, and the metal electrode had

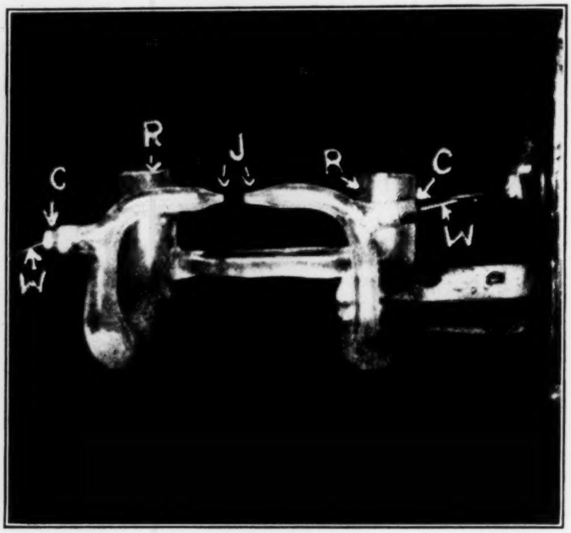

FIG. 1.—Apparatus

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[3] K. B. Thomson and O. S. Duffendack, "A Photographic Method of Measuring the Relative Intensities of Spectral Lines," *Journal*, Optical Soc. America, Vol. 23, p. 101 (1933).

[4] O. S. Duffendack, R. A. Wolfe and R. W. Smith, "Quantitative Analysis by Spectroscopic Methods," *Industrial and Engineerng Chemistry*, Analytical Edition, Vol. 5, p. 226 (1933).

[5] O. S. Duffendack, F. H. Wiley and J. S. Owens, "Quantitative Analysis of Solutions by Spectrographic Means," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 7, p. 410 (1935).

[6] F. Twyman and C. S. Hitchen, "Estimation of Metals in Solution by Means of Their Spark Spectra," *Proceeding*, Royal Soc. (London), Series A, Vol. 133, p. 72 (1931).

to be carefully cleaned before each exposure. In order to avoid the necessity for those precautions, the present authors have developed a source which consists of a spark between two electrodes of the solution to be analyzed.

The apparatus is similar to one described by Lukas[7] and consists of two horizontal jets of quartz or of pyrex glass rigidly joined together. The solution flows through these jets at a very constant rate from reservoirs connected to them. The apparatus may take different forms depending upon the manner in which the reservoirs are connected to the jets and on how the flow of solution is produced and regulated. Perhaps the most satisfactory form of apparatus is that shown in Fig. 1. In this arrangement the jets, *J*, are connected by glass or quartz tubing to reservoirs, *R*, which are bent in the form of an arc of a circle with its center in the axis of the two jets. Electrical connections are made by gold wires passing through rubber caps, *C*, on the jets. The two jets and their reservoirs are rigidly fastened together by quartz or glass rods sealed onto them and the apparatus is then mounted so that it can be rotated about a horizontal axis through the two jets, thus causing the solution to flow from the jets. The rotation is produced by an electric motor connected to the apparatus by suitable gears or pulleys and the rate of flow is determined by the rate of rotation and by the cross-section of the reservoirs. The proper rate of flow is determined by experiment and thereafter kept constant. For the analyses described below, a rate of flow of 1 ml. per min. from each jet was found to be satisfactory. The reservoirs have a diameter of 1.1 cm. and a mean radius of arc of 4 cm. The jets have apertures of 0.75 mm. diameter and are spaced 5 mm. apart.

The spark is excited by a 1-kva. 25,000-v. transformer operated from a 110-v. 60-cycle circuit. The primary current is regulated by suitable controlling rheostats and is kept very constant. For the analyses described a primary current of 8 amp. was found to be satisfactory. No condensers or extra inductances are used in the circuit, and the spark obtained in this way is very arc-like in character and excites only the lowest terms of the arc spectra of the elements in solution.

In a variation of this apparatus, the reservoirs consist of glass cylinders connected to the jets by means of rubber tubing. Flow is then produced by lifting these reservoirs by means of strings wound on a drum rotated by an electric motor. This arrangement is as satisfactory as the other, its only disadvantage being that it requires more solution for an analysis on account of the volume of the connecting tubes. In this arrangement, about 25 ml. are required for an analysis while only 9 ml. are required in the other. A regular rate of flow of the solution from the jets is insured in both cases by using a synchronous motor. The reservoirs and jets are sufficiently cleaned after use by simply rinsing them thoroughly with distilled water.

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[7] H. Lukas, "Über die Intensitatsverhältnisse in den Spektren von Alkaligemischen und die Möglichkeit einer quantitativen Spektralanalyse dieser Elemente," *Zeitschrift für anorganische und allgemeine Chemie,* Vol. 195, p. 324 (1931).

A number of experiments were carried out to determine the effects of variations in the sparking apparatus. It was found that no important changes in the relative intensities of the spectral lines resulted from changes in the primary current from 7 to 10 amp. The current to be used is limited by the danger of boiling the solution in the jets. When this occurred, the solution ran back into the jets and the spark struck to the glass, ruining the jets. Larger currents can be used with larger jets and higher rates of flow.

Changes in the diameter of the jets also had very little effect on the relative intensities of the lines, although the stability of the spark appeared to be affected somewhat. Changes in the separation of the jets, on the other hand, had a very marked effect on the relative intensities of some of the lines and also on the stability of the spark. The spark became too unstable for use when the jets were separated by more than 5.5 mm. It is, therefore, necessary to fix the jets rigidly at a given distance, and if it should become necessary to replace the jets, it might be found necessary to draw up new working curves for the analyses. When jets separated by less than 4 mm. were used, it was found that the calcium line used in the analyses described below became too weak for satisfactory measurements.

Changes in the rate of flow of the solution also affected the relative intensities of certain lines very strongly. Lines of potassium and calcium were especially sensitive to changes in the rate of flow. Therefore it is necessary to maintain a very constant rate of flow.

HIGH-VOLTAGE ARC SOURCE

It is desirable to have a source by means of which an analysis can be made of only a drop of solution. This is particularly true in the case of certain biological fluids and is sometimes desirable in industrial applications. Several sources of this nature have been investigated and the most convenient and at the same time a very satisfactory source was found to be a high-voltage, low-current, alternating current arc between spectroscopic carbons upon which a drop of the solution to be tested has been evaporated.

The electrodes consist of spectroscopic carbons of the highest purity $\frac{1}{4}$ in. in diameter and about $\frac{1}{2}$ in. long. These electrodes have flat ends and a drop of solution was placed on one end of each of two electrodes and quickly evaporated by dry heat so that there was very little penetration of the solution into the carbon. It was found necessary to remove all organic matter from the solution as the presence of such material caused the solution to be absorbed by the carbons, and erratic results were obtained because of the variation in the degree of absorption of the solution by different carbon electrodes. The lower electrode is firmly clamped in place in an arc holder. The upper electrode is held by a spring clip in the upper jaw of the arc holder. This jaw is movable and when pushed down against a stop the two carbons are pressed together end to end, the upper one sliding in its clip to accommodate itself to the position of the lower one. When the movable jaw of the arc holder is released, a spring moves it upward against a stop which fixes the separation of the electrodes. We have found a separation of 2.5 mm. to be suitable for the analyses described.

The arc is excited by a 60-cycle alternating current from a 3 kva. transformer with its primary in a 110-v. circuit. The secondary e.m.f. is about 1100 v. The arc current is regulated by a number of fixed choke coils and a variable rheostat in the secondary circuit and is run at 2 amp. At this low current the arc will not maintain itself on clean carbons and runs practically entirely on the salt, going out when the salt is exhausted. When weak solutions are to be analyzed, a salt of some element not present in the test material is added to the solution before it is evaporated on the electrodes in order to stablize the arc and permit longer burning. We have found it satisfactory to add 2 per cent of lithium as lithium chloride to the test solution for this purpose. On account of the large e.m.f. in the secondary relative to the voltage drop across the arc, the arc burns very steadily and maintains constant excitation conditions. The spectrum consists of the arc lines and spark lines from the lower terms of the elements present in the solution and the 2478Å line of carbon. This line and the carbon bands are relatively weak, and there is practically no continuous background so that the lines are subject to accurate measurement for relative intensity. This source does not yield quite as high accuracy as the jets.

OTHER SOURCES

Two other sources requiring only a drop of solution have been tested. One consists of a hollow cathode discharge in helium. A drop of the solution was evaporated inside the hollow cathode before it was inserted in the discharge tube. This source is quite inconvenient as the cathode must be removed and cleaned after each test, and the tube must be evacuated and filled with helium each time. These operations require considerable time and care. The spectral lines of the test elements are not any too strong but there is apt to be troublesome background unless considerable care is exercised in purifying the gas and degassing the apparatus.

A high-frequency spark source was also tested. In this source a high-frequency spark was excited between a metal electrode and a bit of filter paper moistened with the solution to be analyzed. The filter paper was placed upon a glass plate which, in turn, lay upon a flat metal electrode. This type of source has been used successfully by Gerlach and Schweitzer,[8] and we find it satisfactory for certain spectral regions, especially below

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[8] W. Gerlach and E. Schweitzer, "Die Verwendung von Hochfrequenz zur Funkenerzeugung; der quantitative Nachweis kleinster Quecksilbermengen," *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 195, p. 255 (1931).

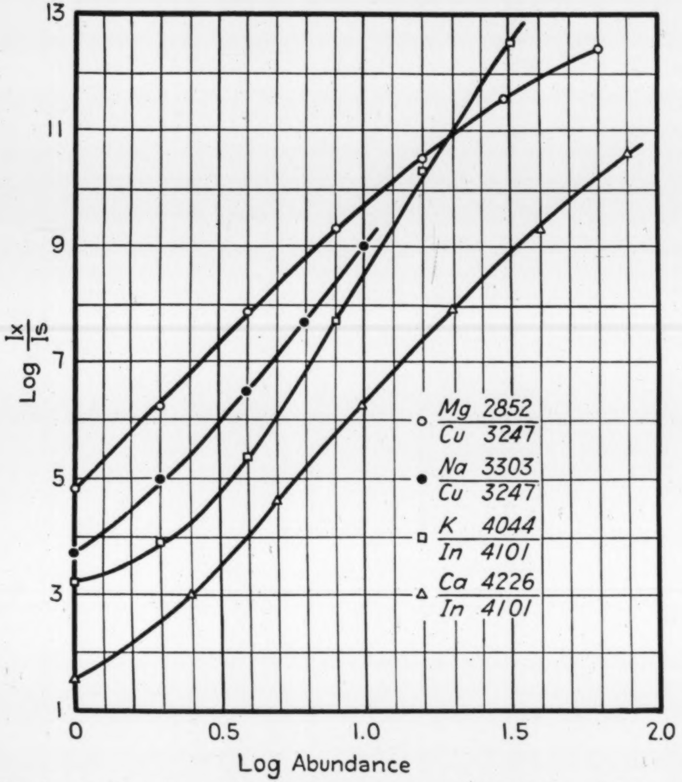

FIG. 2.—Working Curves for the Determination of Magnesium, Calcium, Sodium, and Potassium.

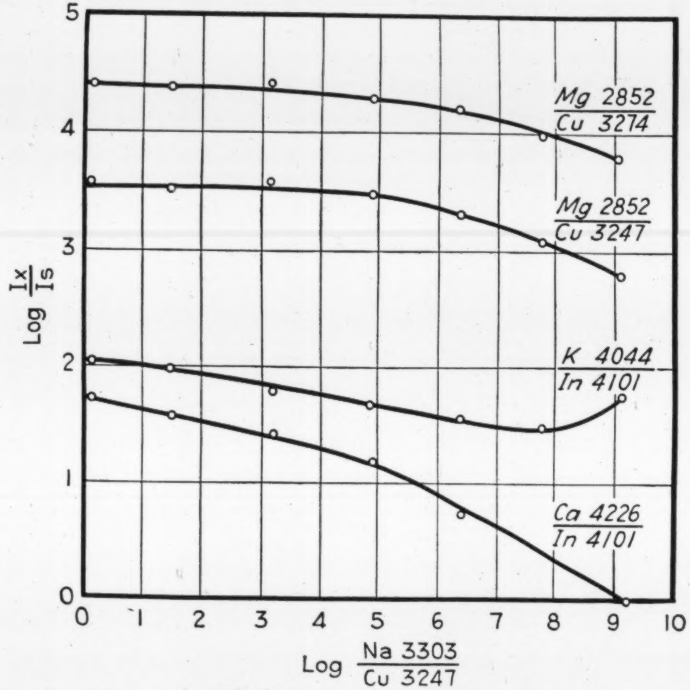

FIG. 3.—Effect of Changes in Sodium Concentration on Relative Intensities of Lines of Other Elements.

2850 Å. The chief difficulty with this source is that it strongly excites the air bands and the water vapor bands and these occupy most of the spectral region above 2850 Å.

TYPICAL RESULTS

The analysis of solutions for sodium, potassium, calcium, and magnesium has been developed especially for the determination of these elements in urine, blood, saliva, and other body fluids. A method for preparing such fluids for analysis has already been described.[5] Typical working curves for these analyses are shown in Fig. 2 in which the log of the ratio of the intensities of selected lines of the test elements and of internal standards is plotted against the log of the percentage of these elements in solution. These curves are straight lines over most of their lengths. The curves of the figure are for a pair of jets like those described above. The curves for the high-voltage carbon arc are very similar and apply to concentrations of the test elements as follows: Mg 0.0005 to 0.05 per cent; Ca 0.002 to 0.10 per cent; Na 0.01 to 1.0 per cent; K 0.05 to 1.0 per cent. The lines of these elements and of the internal standards used in the analyses are shown in the figure. The method has also been applied to the analysis of electroplating solutions for nickel and other elements and of several commercial chemicals produced in quantity.

INFLUENCE OF THE PRESENCE OF OTHER ELEMENTS

That the presence of one element may affect the intensities of the spectral lines of other elements in the same specimen has been demonstrated by other observers,[5,6] and has been studied extensively by the present authors, as such effects must be taken into account in making spectroscopic analyses. Figure 3 shows curves for the effect of varying amounts of sodium on the relative intensities of the lines of other elements present in the solutions. The abscissae are proportional to the concentration of sodium in solution and the ordinates are the logs of the relative intensities of the spectral lines of other elements and internal standards. While it is known that some of these effects are due to energy exchanges between the atoms in the electric discharge, other factors also enter into consideration and it is impossible to predict them or to allow for their influence. They must be discovered and measured and then they can be taken into account or eliminated by one of the methods described by Duffendack, Wiley, and Owens.[5]

ACCURACY AND RELIABILITY

It is important to know to what precision the determination of the amount of a given element may be estimated by any chosen method, and this is all too frequently not well determined. It is essential to know the distribution of errors rather than the average error for one wants to know the probability that a certain determination is correct to within a given percentage error. Figure 4 shows the distribution of errors in the analyses of 97 solutions for sodium and of 76 for potassium by the use of a pair of jets. It may be noted that in no case did the error exceed 10 per cent and errors of less than 1 per cent occurred with the greatest frequency. A peculiar repetition of errors in the determination of potassium of between 3 and 4 per cent is evident from the figure. It has just recently been discovered that this peculiar distribution arises from an error introduced into a group of analyses for potassium by a change in the focus of the spectrograph employed on account of the swelling of its wooden case with an increase in the humidity of the air.

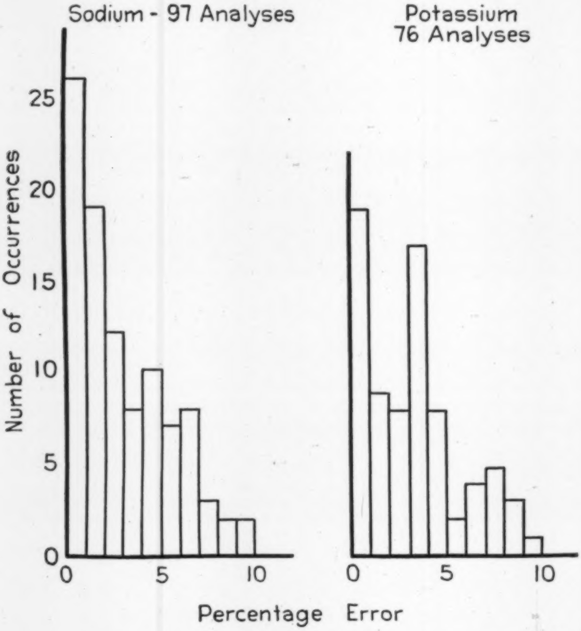

FIG. 4.—Distribution of Errors in Analysis.

One marked advantage in quantitative analyses by such means as those described above lies in the fact that the determination rests upon direct measurements with instruments and at no step in the process is it necessary for the analyst to rely upon his judgment. The analytical procedure may be carried out by one who is not an expert in spectroscopy and reliable results obtained. Tests made of this point with the apparatus described above indicate that determinations made by reasonably skillful laboratory assistants with no special training in spectroscopy are as reliable as those made by spectroscopists of long experience. It is necessary, of course, for an expert to work out the procedure for any given analysis.

Acknowledgments:

This work has been made possible by a grant-in-aid from the Rockefeller Foundation and the authors wish to express their grateful thanks for it. They wish also to express their appreciation of the cooperation of their colleague Dr. W. C. Lee who prepared the specimens for analysis and carried out many chemical tests in conjunction with this investigation.

A HYDROMETER METHOD FOR DETERMINING THE FINENESS OF PORTLAND-PUZZOLAN CEMENTS

BY S. B. BIDDLE JR.[1] AND ALEXANDER KLEIN[1]

SYNOPSIS

The determination of fineness of portland-puzzolan cements involves certain considerations not encountered in the testing of portland cements because of differences in physical properties of the portland-cement component and the puzzolanic component of a portland-puzzolan cement.

To supplement the common methods of mechanical analysis of portland cement and to assist in extending the use of such methods into the field of portland-puzzolan cement, a hydrometer method of analysis has been adapted from that commonly used in soil analysis. The apparatus is inexpensive and the technique of testing is simple. The method essentially as described herein is being used as one of the acceptance tests for approximately 600,000 barrels of portland-puzzolan cement for Bonneville Dam.

In the paper, the theoretical considerations in particle-size determination by hydrometer are reviewed, the procedure of testing and calculating is described, and typical test results are presented and compared with those obtained by independent methods. The concordance of the test results indicates that the assumptions are reasonable and that the degree of reproducibility is satisfactory for practical purposes.

INTRODUCTION

In order to determine the fineness of portland-puzzolan cements employed in investigations of cement and concrete at the Engineering Materials Laboratory of the University of California, use has been made of a hydrometer method similar to that used in the mechanical analysis of soils. The hydrometer is employed since methods commonly used in determining the fineness of portland cements present difficulties when applied to portland-puzzolan mixtures, in which the portland cement and the puzzolan usually differ in specific gravity and opacity, particularly when the proportions of these components are uncertain.

Other Methods:

The use of the No. 200 sieve (74μ openings) is inadequate as a measure of cement fineness at the higher degrees of fineness now coming into general use. The No. 325 sieve (43μ openings) provides a rough indication of the relative fineness of cements, but the percentage retained is not large enough properly to represent the sample as a whole; further, little indication is obtained of the surface area of the sample. Moreover, in portland-puzzolan cements usually the particles of puzzolan are light and bulky and hence they tend to clog the meshes of the sieve when the standard or "dry" method of sieving is employed. When the sample is sieved by the "wet" method (under a water spray) a satisfactory separation is obtained, but the information obtained from the test is subject to the same limitations as in the method of dry sieving.

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[1] Research Engineer, Engineering Materials Laboratory, University of California, Berkeley, Calif.

The methods now in most general use for determining the fineness of portland cements, in terms of surface area, involve the amount of light intercepted by a sample dispersed in a translucent liquid medium.[2,3] The use of this relation is made practical by the nearly total opacity of portland cement; apparently the tendency of a portland-cement particle to intercept light in proportion to projected area varies little until the particle diameter approaches the wave length of light. On the other hand, the particles of certain puzzolans are highly translucent, and the degree of translucency varies inversely with the size of the particles; this translucency renders the use of photometric methods impracticable except for determinations of relative fineness of a given material. In such operations as the control of grinding of portland-puzzolan cements, in which case the composition of the mixture does not vary, the relatively rapid turbidimeter methods may be utilized, provided that turbidimeter constants have been established by a method independent of optical properties. Under similar circumstances, wet-sieving methods may be utilized. In addition to the hydrometer method described herein, methods independent of optical properties include air separation or elutriation, sedimentation, and microscopic counting. However, with the exception of the microneter,[4] the application of these methods in the field of portland-puzzolan cements has been extremely limited.

Hydrometer Method:

If a sample of cement is uniformly dispersed in a suitable liquid medium and simultaneous observations of elapsed time and of specific gravity (as measured by a hydrometer) are made as the particles settle, the particle-size distribution and surface area of the sample may be calculated. It has been demonstrated that for portland cements the surface area thus obtained is in reasonable agreement with values obtained by independent methods, and that for portland and portland-puzzolan cements the values are reproducible to a satisfactory degree, perhaps ±3 per cent.

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[2] L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

[3] Alexander Klein, "A Suspension Turbidimeter for Determination of Specific Surface of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 303 (1934).

[4] The specific surface of commercial portland-puzzolan cements has been determined by Hubert Woods employing the Riverside microneter, as reported by T. E. Stanton and Raymond E. Davis, "Laboratory and Field Tests of Bayshore Highway Cooperative Investigation," reviewed in *Proceedings*, Am. Concrete Inst., Vol. 31, p. 446 (1935).

The applicability of a hydrometer method to the analysis of granular materials, particularly soils, has been well established. The method described herein has been adapted from methods proposed and employed by Bouyoucos,[5] Casagrande,[6] and others. An application of the method of Bouyoucos is described by Hogentogler and Willis.[7] Willis and Johnston[8] have determined the fineness of portland cements by hydrometer.

THEORETICAL CONSIDERATIONS AND METHOD OF CALCULATION

In the application of the hydrometer method to cements, the diameter of a particle is taken as the diameter of a sphere having the same specific gravity and settling velocity as the particle. The equivalent diameter of the sphere is calculated in accordance with Stokes' law. Specific surface is taken as the surface area, in square centimeters per gram, of cement composed of particles whose diameters are determined as described above.

The specific surface of a cement is calculated from the data obtained by means of a hydrometer test in the following steps:

1. Determination of particle diameters.
2. Calculation of particle-size distribution.
3. Calculation of specific surface.

Particle Diameter:

In the calculation of particle diameter and particle-size distribution from the change with time of hydrometer readings, the following assumptions are made:

1. Stokes' law defines the rate of settlement of particles.

2. The separate particles throughout the sample are homogeneous with respect to specific gravity, and are uniformly dispersed at the beginning of the test.

3. The rate of change in specific gravity at any point in the sedimentation cylinder during a test is small and uniform.

Further, for accurate determinations the following conditions should obtain:

1. The cross-sectional area of the sedimentation cylinder should be large compared with the horizontal cross section of the hydrometer bulb.

2. The concentration of particles in suspension at the start of the test should not be sufficient to increase the specific gravity more than about four per cent over that of the liquid medium.

In actual tests, some of these assumptions and conditions are violated to a greater or less extent. However, test results have revealed the effect of these violations to be slight except in extreme cases.

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[5] G. J. Bouyoucos, "The Hydrometer Method for Making a Very Detailed Mechanical Analysis of Soils," *Soil Science*, Vol. 26, pp. 233–238 (1928).

[6] A. Casagrande, "The Hydrometer Method for Mechanical Analysis of Soils and Other Granular Materials," Unpublished Notes, Massachusetts Institute of Technology (1931).

[7] C. A. Hogentogler and E. A. Willis, "Subgrade Soil Testing Methods," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 693 (1934); see Fig. 17, p. 713.

[8] E. A. Willis and C. M. Johnston, "Mechanical Analysis of Portland Cement by the Hydrometer Method," *Public Roads*, Vol. 15, May, 1934, pp. 76–78.

Stokes' law for the relation between size and rate of settlement of a spherical particle in a liquid medium[9] may be expressed as follows:

$$D^2 = \frac{1.837\ Hu \cdot 10^6}{t(s_p - s_m)} \quad \text{.....................(1)}$$

where D = diameter of particle in microns,
u = viscosity of settling medium in poises,
s_p = specific gravity of particle,
s_m = specific gravity of medium,
H = distance which particle has settled in centimeters, and
t = time required for particle to settle H centimeters, in seconds.

From the assumption of the validity of Stokes' law, it follows that all particles of size D will, after time t has elapsed, have settled a distance H. As it is assumed that all particles are uniformly dispersed at the start of the test, the distribution of particles of size D at a horizontal plane at depth H will be the same at time t as at the start of the test. Likewise, there will be no particles of a size greater than D at plane H, and the distribution of particles smaller than D at plane H will be the same as at the start of the test. Hence the specific gravity R of the suspension at depth H and time t is a measure of the concentration of all particles of size D and less at the start of the test.

Particle-Size Distribution:

To determine the proportion of the original sample that is composed of particles of size D or less, the procedure is as follows:

Let W = total weight of all particles of size D or smaller in the sample, and
V = total volume of the suspension.

Then the weight of solid matter per unit volume of the suspension at depth H and time t is $\frac{W}{V}$. The volume of liquid displaced by this material is $\frac{W}{s_pV}$ and the weight of the liquid per unit volume of the suspension is $s_m\left(1 - \frac{W}{s_pV}\right)$. The specific gravity R of the suspension may therefore be written

$$R = \frac{W}{V} + s_m\left(1 - \frac{W}{s_pV}\right) = s_m + \left(\frac{s_p - s_m}{s_p}\right)\frac{W}{V} \quad \text{.........(2)}$$

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[9] Horace Lamb, "Hydrodynamics," Fifth Edition, p. 567, Cambridge University Press, London—New York City (1930).

Charles D. Hodgman and Norbert A. Lange, "Handbook of Chemistry and Physics," Sixteenth Edition, p. 1277, Chemical Rubber Co., Cleveland, Ohio (1931).

and, solving for W,

$$W = (R - s_m)\left(\frac{s_p}{s_p - s_m}\right) V \qquad (3)$$

For convenience in plotting the particle-size distribution of a material W is usually expressed as a percentage $W_{\%}$ of the total weight W_o of the test sample, or

$$W_{\%} = 100 \frac{W}{W_o} = \frac{100}{W_o} (R - s_m)\left(\frac{s_p}{s_p - s_m}\right) V \qquad (4)$$

For a sedimentation cylinder the cross-sectional area of which is large compared with that of the hydrometer bulb, the maximum particle diameter

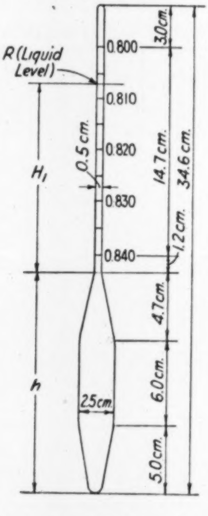

FIG. 1.—Hydrometer.

D for the weight W can be calculated by taking H as the vertical distance from the surface of the liquid to the center of the hydrometer bulb and substituting in Stokes' law (Eq. 1) this value of H and the time t corresponding to the weight W. However, in the narrow sedimentation cylinders which, for practical reasons, must be used in this work, the depth of immersion of the center of volume of the hydrometer bulb is not exactly equal to the distance of settlement H in Eq. 1, because of the relative movement of that portion of the suspension in which the bulb floats when the hydrometer is inserted.

For a given hydrometer and sedimentation cylinder, the value of H for any depth of immersion of the hydrometer (Fig. 1) can be calculated in the following manner:

Let h = length of hydrometer bulb,
h_o = length occupied by the portion of the suspension in which the hydrometer floats, before the hydrometer is immersed,
H_1 = depth of immersion of top of hydrometer bulb,
V_1 = volume of hydrometer bulb,
A = cross-sectional area of sedimentation cylinder, and
H = effective depth of immersion, equal to the distance which particle has settled (Eq. 1).

Then

$$h_oA = hA - V_1$$

or

$$h_o = h - \frac{V_1}{A}$$

As

$$H = H_1 + \frac{h_o}{2}$$

then

$$H = H_1 + \frac{1}{2}\left(h - \frac{V_1}{A}\right) \qquad (5)$$

Using Eqs. 1, 4, and 5, the percentage by weight of the original sample of particles smaller than a given size in a suspension may be determined by observing simultaneously (1) the specific gravity of the suspension by means of the hydrometer and (2) the time which has elapsed since the particles began to settle. A number of observations taken at selected times make it possible to draw a curve which represents the particle-size distribution of the cement.

Calculation of Specific Surface:

From the curve of particle-size distribution, the surface area per gram of cement of the fraction of the sample falling between any two sizes can be calculated in the following manner:

Let D_1 and D_2 represent two particle sizes, in microns, for which $W_{\%1}$ and $W_{\%2}$ are respectively the percentages by weight of the sample finer than D_1 and D_2. Then the average size $\overline{D}$ of this fraction is

$$\overline{D} = \frac{D_1 - D_2}{2}$$

and the percentage $\Delta W_{\%}$ which has this average size is

$$\Delta W_{\%} = W_{\%_1} - W_{\%_2}$$

The volume of a spherical particle of diameter $\overline{D}$ is $\frac{\pi\overline{D}^3}{6}$.

The weight, in grams, of a spherical particle of diameter $\overline{D}$ microns is

$$s_p \frac{\pi\overline{D}^3}{6} \cdot 10^{-12}$$

Hence if ΔW is the total weight, in grams, of particles between the selected sizes, then the number of particles is

$$\frac{\Delta W}{s_p \cdot \frac{\pi\overline{D}^3}{6} \cdot 10^{-12}} \quad \text{or} \quad \frac{6\Delta W \cdot 10^{12}}{\pi s_p \cdot \overline{D}^3}$$

The surface area, in square centimeters, of a spherical particle of diameter $\overline{D}$ microns is $\pi\overline{D}^2 \cdot 10^{-8}$. Hence the total surface area ΔS_t, in square centimeters, of the selected fraction is

$$\Delta S_t = \pi\overline{D}^2 \cdot 10^{-8} \cdot \frac{6\Delta W \cdot 10^{12}}{\pi s_p \cdot \overline{D}^3} = \frac{6\Delta W \cdot 10^4}{s_p \cdot \overline{D}}.$$

When W_o = total weight of sample in grams,

$$\Delta W_{\%} = 100\,\frac{\Delta W}{W_o}$$

then

$$\Delta S_t, \text{ in sq. cm.} = \frac{6}{s_p \cdot \overline{D}} \cdot \frac{\Delta W_{\%} \cdot W_o}{100} \cdot 10^4$$

and

$$\Delta S \text{ (surface area of fraction per gram of sample)} = \frac{\Delta S_t}{W_o}$$

$$= \frac{600\Delta W_{\%}}{s_p \cdot \overline{D}} \text{ sq. cm. per gram of sample} \ldots (6)$$

The summation of the values of ΔS over the entire range of sizes included in the sample is the specific surface S of the sample, expressed in square centimeters per gram.

As in the case of other sedimentation methods in which the specific surface of a cement is calculated from its particle-size distribution, a sufficiently precise relative result is obtained if the specific surface S represents the summation of approximately ten values of ΔS distributed over the particle-diameter range of 8 to 100μ, plus a value of ΔS obtained by means of a suitable assumption regarding the average diameter of all particles smaller than 8μ. In this work it has been assumed that these smallest particles have an average diameter of 5.35μ; the basis for this assumption is discussed in the Appendix.[10]

APPARATUS AND PROCEDURE

With the exception of the hydrometer itself, the apparatus required for this test is simple and of a type found in any cement laboratory. The hydrometer used has a range of specific gravity 0.80 to 0.84. The dimensions of the hydrometer are given in Fig. 1. Kerosine containing approximately 2 per cent by volume of oleic acid is used as a liquid medium. 1000-ml. graduates serve as sedimentation cylinders.

The test procedure is briefly as follows: A sample for test consists of 40.00 g. of cement taken from a quantity which has been passed through a No. 100 sieve and then (unless freshly ground) dried in an oven maintained at 110 C. The test sample is placed in a beaker with 200 ml. of kerosine and 20.0 ml. of oleic acid, Technical (red oil), thoroughly mixed, and raised to a temperature of 150 C. The sample is then allowed to cool to room temperature, again thoroughly mixed for at least five minutes by means of an electrically driven mechanical stirrer, and placed in a 1000-ml. graduate which is then filled to the 1000-ml. mark with kerosine.

The sample is dispersed in the liquid by closing the open end of the graduate with one hand and alternately inverting and righting the graduate, for a period of 1 min. (or longer if more time is required to place all the material in suspension). When the graduate is righted for the last time, it is placed on a vibration-free level table; this time is the starting time of the test.

At 1, 5, 15, 30, 45, 60, 75, and 90 min. after the test is started, the specific gravity is measured by means of the hydrometer. The hydrometer is slowly immersed $\frac{1}{2}$ min. before each reading is taken, and is slowly withdrawn immediately after each reading, allowing 5 sec. for insertion or withdrawal. Care is taken to avoid disturbance during the period of test. Either the test is made in a room maintained at constant temperature or the sedimentation cylinder is placed in a water bath to reduce temperature variations to not greater than ±0.5 C.

The particle-size distribution and specific surface of the sample are calculated as previously described and as indicated in the example of Fig. 2, employing the data listed below. These data are measured with the precision indicated

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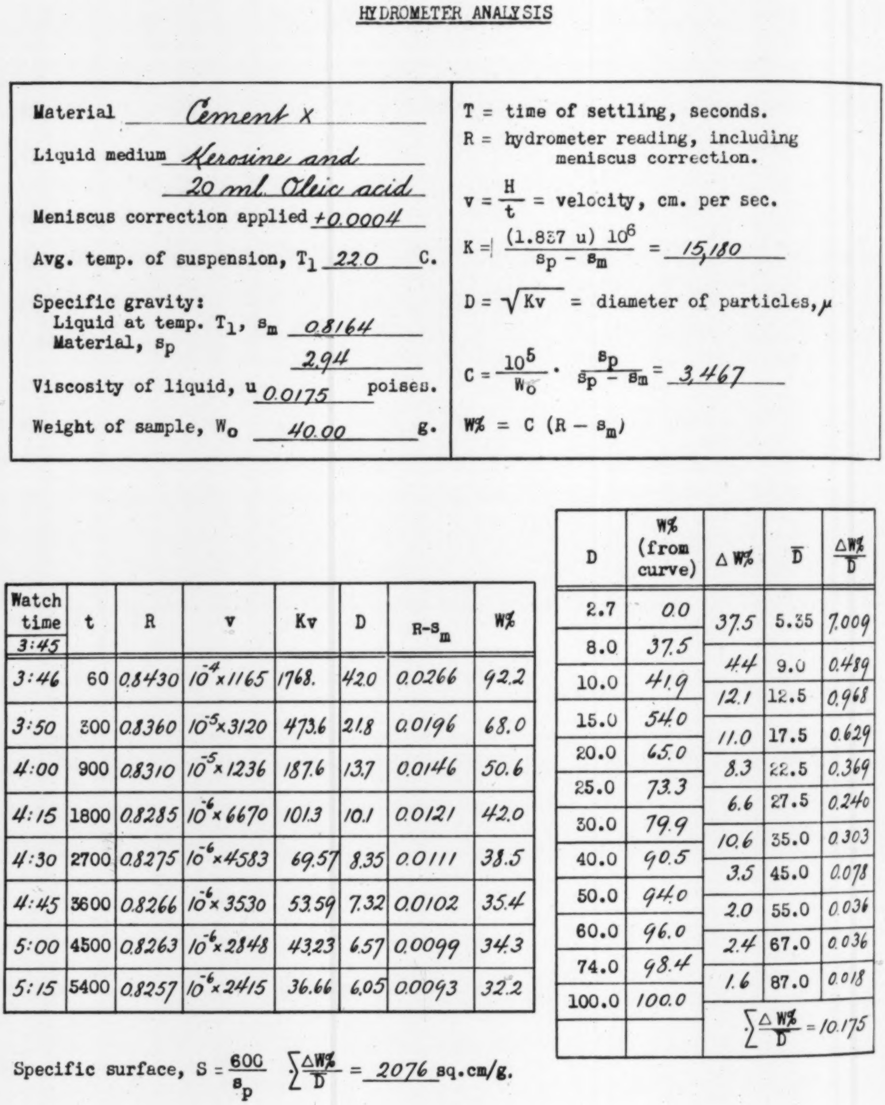
[10] See p. 323.

(*a*) Specific gravity R at the stated times, determined by observation with the hydrometer (± 0.0001).



HYDROMETER ANALYSIS

Material Cement X

Liquid medium Kerosine and 20 ml. Oleic acid

Meniscus correction applied +0.0004

Avg. temp. of suspension, T_1 22.0 C.

Specific gravity:

Liquid at temp. T_1, s_m 0.8164

Material, s_p 2.94

Viscosity of liquid, u 0.0175 poises.

Weight of sample, W_o 40.00 g.

T = time of settling, seconds.

R = hydrometer reading, including meniscus correction.

$v = \frac{H}{t}$ = velocity, cm. per sec.

$K = \frac{(1.837\ u)\ 10^6}{s_p - s_m}$ = 15,180

$D = \sqrt{Kv}$ = diameter of particles, μ

$C = \frac{10^5}{W_o} \cdot \frac{s_p}{s_p - s_m}$ = 3,467

$W\% = C\ (R - s_m)$

Watch time 3:45	t	R	v	Kv	D	$R-s_m$	W%
3:46	60	0.8430	$10^{-4} \times 1165$	1768.	42.0	0.0266	92.2
3:50	300	0.8360	$10^{-5} \times 3120$	473.6	21.8	0.0196	68.0
4:00	900	0.8310	$10^{-5} \times 1236$	187.6	13.7	0.0146	50.6
4:15	1800	0.8285	$10^{-6} \times 6670$	101.3	10.1	0.0121	42.0
4:30	2700	0.8275	$10^{-6} \times 4583$	69.57	8.35	0.0111	38.5
4:45	3600	0.8266	$10^{-6} \times 3530$	53.59	7.32	0.0102	35.4
5:00	4500	0.8263	$10^{-6} \times 2848$	43.23	6.57	0.0099	34.3
5:15	5400	0.8257	$10^{-6} \times 2415$	36.66	6.05	0.0093	32.2

Specific surface, $S = \frac{600}{s_p} \sum \frac{\Delta W\%}{D}$ = 2076 sq.cm/g.

D	W% (from curve)	Δ W%	$\overline{D}$	$\frac{\Delta W\%}{\overline{D}}$
2.7	0.0	37.5	5.35	7.009
8.0	37.5	4.4	9.0	0.489
10.0	41.9	12.1	12.5	0.968
15.0	54.0	11.0	17.5	0.629
20.0	65.0	8.3	22.5	0.369
25.0	73.3	6.6	27.5	0.240
30.0	79.9	10.6	35.0	0.303
40.0	90.5	3.5	45.0	0.078
50.0	94.0	2.0	55.0	0.036
60.0	96.0	2.4	67.0	0.036
74.0	98.4	1.6	87.0	0.018
100.0	100.0			
		$\sum \frac{\Delta W\%}{\overline{D}} = 10.175$		

FIG. 2.—Typical Calculations.

(*b*) Average temperature T_1 of the suspension, determined by observations during the test ($\pm$ 0.5 deg. Cent.).

(*c*) Specific gravity s_m of the liquid medium (kerosine and oleic acid) at the temperature of the test, as measured by the hydrometer ($\pm$ 0.0001).

(*d*) Viscosity u of the liquid medium (kerosine and oleic acid) at the temperature of the test, determined by separate test (± 0.0001 poises).

(*e*) Specific gravity s_p of the material, determined on a separate sample by means of the Le Chatelier flask (± 0.01).

(*f*) Weight W_o of the sample of oven-dry material (± 0.01 g.).

(*g*) Effective depth H of hydrometer immersion (depth of particle settlement), computed from Eq. 5 (± 0.02 cm.).

For each hydrometer reading R is calculated the percentage $W_{\%}$ of the sample finer than a diameter D which is calculated in accordance with Stokes' law. These data are plotted on 2-cycle semi-logarithmic paper, with percentages as the ordinates to natural scale, and particle diameters in microns as the abscissas to logarithmic scale. From the curve connecting these plotted points are tabulated the corresponding values of the percentage ($W_{\%}$ from curve) by weight of particles having diameters less than 8, 10, 15, 20, 25, 30, 40, 50, 60, and 74μ. The surface area (per gram of sample) of the fraction which falls between each pair of adjacent diameters and above and below the extreme sizes is calculated from the percentage $\Delta W_{\%}$ and average diameter $\overline{D}$, under the assumptions that (1) the average diameter $\overline{D}$ of particles in each fraction is equal to the mean of the two limiting diameters, (2) the average diameter $\overline{D}$ of all particles smaller than 8μ is 5.35μ, and (3) the maximum size of particles is 100μ.[11] The sum of these surface areas of the fractions is the specific surface S of the sample. (For convenience in calculation, first the quantity $\dfrac{\Delta W_{\%}}{\overline{D}}$ is determined for each fraction; then $S = \dfrac{600}{s_p} \quad \Sigma \dfrac{\Delta W_{\%}}{\overline{D}}$).

Although a single hydrometer test requires several hours of elapsed time, the actual time per test may be reduced to perhaps one man-hour if several tests are made concurrently. When elapsed time is important, as in the control of grinding, an occasional hydrometer test for calibration and checking purposes makes possible the use of turbidimeter or wet-sieving methods.

The viscosity of the liquid medium may be determined by means of the Ostwald viscometer,[12] the Saybolt Thermo-viscometer, or a simple form of capillary tube.[13] The Ostwald viscometer is inexpensive and may be obtained as stock equipment from any of several manufacturers of scientific apparatus and supplies. The viscosity of a mixture of kerosine and 2 per cent of oleic acid is only about 0.3 per cent greater than that of the kerosine; hence the same value may be used for both. If determinations of cement fineness are to be made under varying temperature conditions, the variation in viscosity of kerosine with temperature should be determined and plotted for a range of temperature; then the test values of viscosity at the various times of observation can be taken from this diagram.

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[11] If all of the material passes some fine sieve, such as the No. 200 or No. 325 sieve, the maximum particle diameter can be established by extrapolating back on a smooth curve to the 100-per-cent ordinate the particle-size-distribution diagram as calculated from 1-min., 2-min., and 5-min. hydrometer readings. If desired, the same procedure may be followed in preference to the assumption of a maximum diameter of 100μ.

[12] Alexander Findlay, "Practical Physical Chemistry," pp. 70–74, Longmans, Green and Co., New York City (1923).

[13] W. H. Walker, W. K. Lewis and W. H. McAdams, "Principles of Chemical Engineering," pp. 79–80, McGraw-Hill Book Co., Inc., New York City (1923).

The specific gravity of a mixture of kerosine and oleic acid differs appreciably from that of the kerosine; and this value must be determined for the temperature or temperatures of test.

A few puzzolanic materials have been encountered which when tested alone (not in a portland-puzzolan mixture) cannot be satisfactorily dispersed in kerosine by the treatment described herein. For these puzzolanic materials a liquid medium composed of water and 10 ml. of a 17.5-per-cent solution of TDA, an organic dispersing agent, has been found satisfactory. A hydrometer having a range of 1.00 to 1.03 is employed. The test procedure is the same except that the solution is not heated.

TABLE I.—REPRODUCIBILITY OF RESULTS.

Material	Specific Surface (by Hydrometer), sq. cm. per g.		Difference	
	First Test	Second Test	Sq. cm. per g.	Per cent
Portland cement R	1314	1280	+34	+2.6
Portland cement S	1410	1375	+35	+2.5
Portland-puzzolan cement T	1821	1808	+13	+0.7
Portland-puzzolan cement U	2053	2068	−15	−0.7
Puzzolan V	2690	2687	+3	+0.1
Average	1858	1844	+14[a]	+0.8[b]

[a] Algebraic average; the arithmetical average is 20 sq. cm. per g.

[b] Algebraic average; the arithmetical average is 1.3 per cent.

TEST RESULTS

Table I indicates the degree of reproducibility obtained by the hydrometer method. The tests reported in this table comprise five duplicate determinations made by an operator who had had no previous experience with the method. The samples include two commercial portland cements, two commercial portland-puzzolan cements, and a ground puzzolanic material. It is seen that the maximum difference between duplicate determinations is 2.6 per cent, and that the average difference is 0.8 per cent. This degree of reproducibility is typical of that obtained by other operators.

In Table II are given values of specific surface of five portland cements of widely varying composition, as determined by means of the hydrometer

TABLE II.—SPECIFIC SURFACE OF SEVERAL PORTLAND CEMENTS, AS DETERMINED BY TESTS WITH A HYDROMETER AND WITH A KLEIN TURBIDIMETER.

Cement	Time of Grinding, hr.	Specific Surface, sq. cm. per g.		Difference, percentage of turbidimeter value
		Hydrometer	Turbidimeter	
X	3.0	1450	1470	−1.4
	4.0	1670	1755	−4.8
	5.0	1860	1925	−3.4
	6.0	2050	2120	−3.3
A	2.6	1790	1810	−1.1
	4.2	2100	2200	−4.5
B	3.8	1790	1805	−0.8
	5.8	2170	2200	−1.4
ML	1.0	1620	1650	−1.8
LL	1.0	1600	1620	−1.2
Average		1810	1855	−2.4

TABLE III.—CALCULATED AND OBSERVED SPECIFIC SURFACE OF PORTLAND-PUZZOLAN CEMENTS OF KNOWN PROPORTIONS.

Ground Calcined Puzzolan			Portland-Puzzolan Cement[a]					
Ground Calcined Description	Specific Gravity	Specific Surface, sq. cm. per g.	Proportions		Specific Gravity (Observed)	Specific Surface		
						sq. cm. per g.		Difference, per cent of calculated value
			Puzzolan	Cement		Calculated	Observed	
Diatomaceous shale	2.32	2380	10	90	3.07	1800	1810	+0.6
			20	80	2.99	1870	1790	−4.3
			25	75	2.95	1900	1860	−2.1
			30	70	2.91	1930	1930	0.0
			45	55	2.78	2030	2020	−0.5
Granite	2.64	2050	10	90	3.10	1770	1740	−1.7
			20	80	3.06	1800	1660	−7.8
			30	70	3.00	1830	1840	+0.5
			45	55	2.91	1880	1900	+1.1
Pumicite	2.37	2950	20	80	3.00	1980	1840	−7.1
			30	70	2.87	2100	2040	−2.9
Clay A	2.64	1890	20	80	3.06	1770	1640	−7.3
Clay C	2.54	2440	20	80	3.04	1880	1760	−6.4
Diatomaceous earth	2.24	2550	20	80	2.98	1900	1850	−2.6
Average						1889	1834	−2.9[b]

[a] Mechanical mixture (not interground) of puzzolan and high-lime portland cement in the proportions indicated. Specific surface of portland cement 1740 sq. cm. per g.; specific gravity of portland cement 3.16.

[b] Algebraic average; the arithmetical average is 3.2 per cent.

and by means of the Klein turbidimeter. One of the cements is ground to four degrees of fineness, and each of two others is ground to two degrees of fineness. It is seen that the hydrometer results are all lower than those obtained with the turbidimeter, the average difference being 2.4 per cent. The hydrometer values would also show close agreement with values obtained by the use of the microneter of the Riverside Cement Co.; in the microneter method the same assumption is made regarding the average diameter of particles smaller than 8μ. The agreement with the results obtained by means of the Klein turbidimeter is especially significant as the method employing this turbidimeter is independent of sedimentation and the attendant assumptions.

Table III summarizes the results of a series of hydrometer tests designed to indicate the effect of the presence of two materials of different specific gravity in the suspension. Mechanical mixtures were prepared in various proportions, each mixture containing a portland cement and one of six puzzolanic materials for which the specific surface had been determined by hydrometer analysis. The specific surface of these mixtures was determined by means of the hydrometer, and was also calculated from the proportions and fineness of the ingredients. It is seen that the observed specific surface is generally lower than that calculated from the proportions of the mixture, the average difference being 2.9 per cent. Inasmuch as these differences are affected by the possible errors in three separate tests (on the cement, puzzolan, and mixture), the agreement is considered to indicate that for the cements tested no serious error is caused in a hydrometer method by the assumption that such a mixture or an interground portland-puzzolan cement is homogeneous with respect to the specific gravity of the particles. However, for coarse portland cements blended with fine puzzolans the agreement would probably not be so close.

CONCLUDING REMARKS

The hydrometer furnishes a simple, dependable, and fairly accurate means of determining the fineness of portland-puzzolan cements, for which determination the methods commonly employed for portland cements are subject to limitations. The equipment required is inexpensive, and little technical skill is needed for its operation. It is believed that the assumptions are reasonable, and that the values represent the property of fineness of cement which is reflected in such tests as those for strength and heat of hydration. The hydrometer method is being used for the determination of specific surface in acceptance tests of portland-puzzolan cement (approximately 600,000 bbl.) for Bonneville Dam; the tests are made by the San Francisco laboratory of the National Bureau of Standards.

APPENDIX

PARTICLE-SIZE DISTRIBUTION BELOW 8μ

Particle-size distribution can be described by the maximum size, the minimum size, and the percentages of a number of intermediate sizes. The surface area of large particles is relatively small, and for cements the large particles are relatively few and therefore not of great significance; herein the maximum size of all material passing the No. 100 sieve has been taken arbitrarily as 100μ. The particle-size distribution from 100μ to 8μ is determined by means of hydrometer observations at several increments of sedimentation time, as previously described. The distribution below 8μ and approaching the minimum limiting size may be similarly observed, but because of the extended time required for sedimentation of these small particles direct observation is not practicable for routine tests.

Table IV.—Analysis of Material Recovered from Suspension After Hydrometer Testing.

	Portland Cement C-1200	Portland-Puzzolan Cement CP-1900
Specific surface, sq. cm. per g.	1240	1900
Time of settling, *t*, hr.	20	36
Calculated maximum size retained in suspension after time *t*, μ	2.0	1.5
Percentage of original sample retained in suspension after time *t*	0.6	1.2
Oxide analysis of material retained in suspension:		
Percentage of CaO	76.4	94.3
Percentage of SiO_2	14.7	1.2
Percentage of $Al_2O_3 + Fe_2O_3 + SO_3$	8.9	4.5

Table V.—Specific Surface of Typical Cements, as Calculated Under Various Assumptions Regarding the Limiting Size Below 8μ.

Cement	Specific Surface of Total Sample, sq. cm. per g.			
	By Klein Turbidimeter	By Hydrometer		
		Basis A[a]	Basis B[b]	Basis C[c]
C-2200	2220	2060	2220	2540
C-1200	1170	1240	1190	1490
CP-1900	....	1900	1900	2275

[a] Basis A: Assuming average particle size of 5.35μ, as descrbed herein.

[b] Basis B: Employing observed distribution from 2.0 to 8μ.

[c] Basis C: Assuming average particle size of 4.0μ, in accordance with A.S.T.M. Tentative Method (C 115 - 34 T).

By hydrometer, the particle-size distribution below 8μ was determined for (1) a typical coarse portland cement C-1200, (2) a typical fine portland cement C-2200, and (3) a commercial portland-puzzolan cement CP-1900. The observations were continued until sedimentation ceased (20 hr. for C-1200 and C-2200, 36 hr. for CP-1900). The maximum size of the material remaining in suspension was calculated, from Stokes' law and the observed sedimentation interval, to be about 2.0μ. The analysis of the suspended material is shown for the two types of cement in Table IV. It appears that the particles below 2μ may be neglected in computing the specific surface of a normally ground cement because (1) the percentage is very small; (2) the chemical analysis indicates that the material consists chiefly of free lime, gypsum, and decomposition products resulting from the action of oleic acid; and (3) it may be reasonably assumed that any portland-cement particles are already hydrated to a large degree and therefore that with respect to strength their surface area is not significant.

Figures 3 and 4 show the observed particle-size distribution between 2 and 8μ. It is seen that the distribution is not uniform. A similar distribution of particle size within this range has been observed by Willis and Johnston.[14]

For testing purposes, it is highly desirable to assume some uniform distribution below 8μ, such that the calculated surface area of the fraction will approximate that obtained by direct observation. In the work with which the authors have been connected, it has been assumed that the material varies uniformly from 2.7 to 8μ and that the average particle diameter is 5.35μ; this assumption was originally adopted by the Riverside Cement Co. from a consideration of microscope data obtained in the development of its microneter method. In Figs. 3 and 4 it is seen that the particle-size distribution under such an assumption approaches more closely to the observed distribution than does the particle-size distribution assumed to vary uniformly below 8μ with an average particle diameter of 4μ, essentially as embodied in A.S.T.M. Tentative Method of Test for Fineness of Portland Cement by means of the Turbidimeter (C 115 – 34 T).[15]

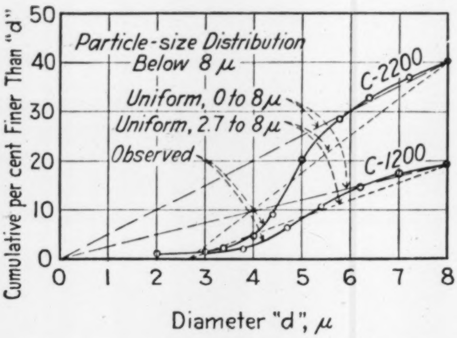

FIG. 3.—Particle-Size Distribution Below 8μ for Typical Portland Cements.

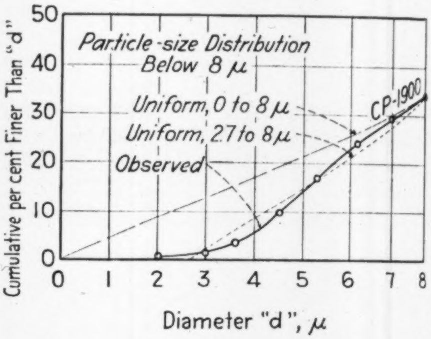

FIG. 4.—Particle-Size Distribution Below 8μ for a Typical Portland-Puzzolan Cement.

A further comparison of the two assumptions is afforded by the data of Table V. The assumption that the average diameter of particles below 8μ is 5.35μ results in values of specific surface of total sample which compare favorably with the observed values; but the assumption embodied in A.S.T.M. Tentative Method C 115 – 34 T results in values of specific surface of total sample which differ considerably from the values observed directly by hydrometer.

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[14] E. A. Willis and C. M. Johnston, "Mechanical Analysis of Portland Cement by the Hydrometer Method," *Public Roads*, Vol. 15, May, 1934, pp. 76–78.

[15] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 777 (1935); also 1935 Book of A. S. T. M. Tentative Standards, p. 432.

DISCUSSION

Mr. J. C. Pearson.[1]—This paper will undoubtedly have considerable reference value in the *Proceedings* of the Society, although much of it is not new. My chief criticism of it is that it conveys the impression that little technical skill is required to use the hydrometer test and obtain with it very excellent and dependable results. From my own experience with the method, I would not belittle the fineness of the technique required, nor would I have quite all the confidence of the authors in its indications. It is the sort of test that savors more of physics than engineering, requiring critical attention on the part of the operator. The reproducibility and dependability of such a test can hardly be determined by a few tests in a single laboratory.

On the other hand, if we do not worry at all about such questions as the applicability of Stokes' law to a relatively heavy concentration, the long-time effect of an acid deflocculent on an alkaline material, the disturbing effect of repeated immersions of the hydrometer, and the lack of sensitivity to small density changes toward the end of the test, then we realize that the method at least has some possibilities of extending our knowledge of particle size distribution in the finer fractions of portland cement.

Mr. W. C. Hanna[2] (*presented in written form*).—Although our laboratory has not investigated this method extensively, considerable work involving its use has been done in the past. Our experience has shown that in ordinary routine work we could not expect the high degree of reproducibility shown by the authors. We would expect the probable error of a single observation to fall at between 4 and 5 per cent in the range near 2000 sq. cm. per g. However, this is not a serious criticism because it is granted that this probable error could be decreased considerably by adopting certain refinements in the procedure.

Our objection to the method as a plant control method or a specification test is that the time consumed to obtain a result is far greater than is allowable. Readings must be taken over a period of 1½ hr., to which is added the time necessary for calculations. If this method is carried out in accordance with the procedure outlined in Paragraph 237 of the Specifications for Portland-Puzzolan Cement for Bonneville Dam, 3 hr. are required for drying the sample besides the time required for sieving, mixing and heating with kerosine and other preparations. We found that at the very least 5 hr. would lapse before results could be obtained and it is doubtful that the average time for routine work could be as low as this.

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[1] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.
[2] Chief Chemist, California Portland Cement Co., Colton, Calif.

It is particularly gratifying that the authors have presented the question as to the minimum size of particles to be expected in cements as given in the appendix to the paper. During an investigation concerned with other work our laboratory obtained data which made it possible to estimate the practical minimum size of particles present. This proved to be 1.8 μ which checks well with the results shown by the authors. We believe it is very important that a truly scientific figure be adopted for this lower limit.

We would agree that 5.35 μ would be a logical average diameter for the smallest fraction in the light of present knowledge.

MR. J. R. GRAN.[3]—There are two details in this test that seem to me to be objectionable from a theoretical standpoint. First, the hydrometer bulb is introduced into and removed from the suspension no less than eight times, which must result in a certain amount of agitation and, to an unknown extent, change the actual time of settling of the particles from the computed time. Second, the hydrometer does not measure the specific gravity of the suspension at any one level, but instead an integration of a varying specific gravity of a column of the suspension of approximately 16 cm. total depth. This could possibly be taken care of by the proper mathematical interpretation of the readings; but to illustrate the results obtained by the proposed method of calculation, suppose the charge consisted entirely of 10 μ material. With other apparatus used for specific surface measurements we could determine that the material was all very close to the 10 μ size; with this apparatus and by this method, however, the charge would appear as a fraction ranging from about 8 to over 40 μ. Since specific surface is calculated by a summation of several fractions, the final result is no more reliable than the measurement of the individual fractions.

In regard to the use of 5.35 μ as the surface mean diameter of the 0 to 8μ fraction, that, to my mind, is open to question. Sedimentation methods are not applicable for the measurement of particles of this size range because it is difficult, if not impossible, to keep the particles properly dispersed for anything like the period required. Conglomeration of cement particles in an oleic acid - kerosine suspension can be detected within a few minutes after agitation ceases. This phenomenon is undoubtedly the explanation of the trend of the curves rather than the grading of the cement.

MR. RAYMOND WILSON[4] (*presented in written form*).—This paper is a welcome addition to the literature on methods of measuring grading in the size ranges beyond the reach of sieves. The low cost of the apparatus is a particularly attractive feature. Apparently the hydrometer method is capable of reasonable accuracy in the determination of weight distribution. As a means of calculating surface area it is subject to question, the doubt arising almost entirely from the uncertainty regarding the actual distribution of the material in the fraction smaller than the smallest separation size included in the observations. Inasmuch as the finest fraction usually accounts for a majority of the total surface area, results by different methods of test depend primarily on identity in the assumptions regarding distribution within that fraction.

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[3] Assistant Materials Engineer, Cement Reference Laboratory, National Bureau of Standards, Washington, D. C.

[4] Conservation Engineer, Portland Cement Assn., Chicago, Ill.

TABLE I.—MICROSCOPE COUNTS ON PORTLAND CEMENTS.

Slides made from No. 0 to No. 200 cement.
Count made at 1000 diameters magnification.
Size range in each interval is 1μ; for example, particles from 4.5 to 5.5 are counted as 5μ.

Size, μ (d)	Normal Cement			High-Early-Strength Cement		
	Number of Particles (f)	fd^2	fd^3	Number of Particles (f)	fd^2	fd^3
1	1305	1305	1 305	2505	2505	2 505
2	268	1072	2 144	294	1176	2 352
3	92	828	2 484	131	1179	3 537
4	66	1056	4 224	72	1152	4 608
5	46	1150	5 750	42	1050	5 250
6	27	972	5 832	16	576	3 456
7	19	931	6 517	14	686	4 802
8	16	1024	8 192	9	576	4 608
Total		8338	36 448		8 900	31 118
	Average Size $\frac{(\Sigma fd^3)}{(\Sigma fd^2)}$	4.4			3.5	

TABLE II.—AVERAGE PARTICLE SIZE OF 0 to 8μ FRACTION OF VARIOUS PULVERIZED CLINKERS.

MATERIAL	AVERAGE PARTICLE SIZE OF 0 TO 8 FRACTION
Cement from large compartment mill	3.8
Cement from same plant ground closed circuit in smaller compartment mill	3.7
Products of successive compartments of a 4-compartment mill:	
First compartment	4.5
Second compartment	4.8
Third compartment	4.3
Fourth compartment	4.2
Two cements ground open circuit in same tube mills (Table I):	
Normal cement (surface = about 1500)	4.4
High-early-strength cement (surface = about 2500)	3.5

Reasonably exact observation of distribution below 8 or 10 μ is entirely too tedious to incorporate into a routine test. It is therefore customary to adopt some convention regarding the distribution within the finest fraction. One generally used convention is that of uniform distribution of *surface* below the smallest separation size, equivalent to using one-half the upper limiting size of the fraction as the mean diameter of the particles in the fraction. The convention of uniform weight distribution is not used in this connection, inasmuch as the indicated surface is infinite when this convention is applied rigidly.

The authors have used 5.35 μ as the mean diameter of the 0 to 8 μ fraction. Their selection of this size was based on prolonged sedimentation—20 to 36 hr. Their conclusion relative to surface area of the fine fraction is quite different from that reached by the writer from direct microscopic observation on a number of cements and on intermediate products of clinker grinding. The results of microscopic counts on a normal and on a high-early-strength cement ground in the same plant are given in the accompanying Table I. Table II gives the average particle size computed from similar tests on several products. These tests include a size range from 0 to 8.5 μ, with an average particle size of 4.25 μ by the uniform surface distribution convention. From the instances cited, it seems likely that the actual average size will generally tend to be less than the size indicated by this convention, especially in fine cements. There are possibilities of substantial errors in the microscopic examinations, but the directness of the method entitles it to at least equality in reliability with a long sedimentation test.

In the absence of specific information some convention has to be used in any surface calculation based on weight distribution. Table II indicates that the convention and the actuality will seldom agree exactly. However, the uniform surface distribution convention is apparently a closer approach than that used by the authors of the paper.

The value for average size used by the authors cannot be criticized so long as it is recognized as being a convention, designed as a substitute for definite information which is hard to get. However, both the Klein and the Wagner turbidimeters "see" the differences in gradation in the finest fraction of cement, though the usual method of calculating Wagner observations fails to take full cognizance of this fact. If the convention used in weight-surface calculations represents the actuality closely, the results will agree well with those obtained by the turbidimeter. The data cited in Table V of the paper are confirmation of this view, if the turbidimeter calibration involved the use of the assumption that the average size in the 0 to 8 μ fraction was 5.35 μ.

Neglecting the material finer than 2 μ seems hardly justifiable. It makes a substantial contribution to the actual surface, and to the surface seen by the turbidimeter. At the present time its effect on cement properties cannot be considered as negligible. Until these points have been cleared up, it appears desirable to use the arbitrary convention of uniform surface distribution in the fine fraction. This convention is preferable to that suggested by the authors since it appears from direct experimental observation to average closer to the truth than any convention assigning a higher average particle size to the finest fraction. It therefore has a better chance to give results in agreement with other methods of test, and, by lessening the differences between methods, to increase confidence in surface area figures.

MR. P. S. ROLLER.[5]—Particle-size distribution, it should be recognized, is as definite a physical property of a material as its density. Density conceivably might be measured by determining dielectric constant and refractive index. But how much reliance could one place in a result obtained in this way, as compared to a direct determination by means of the pycnometer? It occurs to the writer that measurement of particle size frequently has been attempted by methods hardly more far-fetched than that of a dielectric constant measurement for density.

The most reliable methods for measuring size distribution consist in direct observation or in isolation of the particles. The microscope is the basis for the first method and Stokes' law of particle fall, which has been amply confirmed, is the basis for the second. To make use of other incompletely known factors would lead inevitably to an unreliable result. I am thinking particularly at this time of the introduction of the complicated factor of turbidity.

In the writer's opinion the use of the Bouyoucos hydrometer also includes a factor of great theoretical and practical difficulty. Bouyoucos originally used his hydrometer as a means of estimating colloids (nonsettling material) in soils. Subsequent attempt by others to employ it in quantitative measurement of particle size has been criticized severely. One need mention only that theory demands that the density of the suspension be measured in a horizontal place, yet Biddle and Klein use a hydrometer bulb that extends over half the total depth of suspension. Such a procedure has never been logically justified, and practice has shown how very precarious it is.

Much of the authors' paper is concerned with matters already fairly well known. The important problem of deflocculation has not been solved satisfactorily. It would be desirable to compare the effectiveness of various deflocculants and to prove which is best and under what conditions each is to be used.

A few words may be said about the authors' method of calculating surface area as a summation of weight per cent over average diameter. The surface area thus obtained may be called an increment value that is to be contrasted with the true integral value. The writer has discussed this matter in some detail in a paper to be published shortly.[6] It is shown there that if the finest fraction is 0 to 5 μ, the increment or integral values agree for a surface area of 2000 sq. cm. per g. or less. For finer materials the increment value is too low; and indeed may be remarkably so. The explanation of this, of course, is to be found in the fact that the increment surface area has a categorical upper limit that is defined by the average size of the finest fraction. Where the increment surface area calculates out to be greater than 2000 sq. cm. per g., the result should not be accepted without reserve.

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[5] Associate Chemist, U. S. Bureau of Mines, New Brunswick, N. J.

[6] P. S. Roller, "Law of Size Distribution and Statistical Description of Particulate Materials," to be published in the *Journal* of the Franklin Inst.

MR. P. H. BATES.[7]—I should like to call attention to the last sentence in the paper, which reads: "The hydrometer method is being used for the determination of specific surface in acceptance tests of portland-puzzolan cement (approximately 600,000 bbl.) for Bonneville Dam; the tests are made by the San Francisco laboratory of the National Bureau of Standards." That is correct; the specifications were prepared under the supervision of the U. S. engineers and the Bureau of Standards was requested to make the tests according to specifications. The Bureau is making the tests according to the specifications, but in so doing it does not in any way indicate the Bureau's attitude towards the hydrometer as a method for determining fineness.

MESSRS. S. B. BIDDLE, JR.[8] AND ALEXANDER KLEIN[8] (*authors' closure by letter*).—The preceding discussions, while not entirely in agreement with the conclusions drawn in the paper as presented, are gratifying in that they indicate active interest in the problem. For the most part the criticisms are offered on an entirely reasonable basis, yet it should be pointed out that the basis of the criticisms, other than those dealing with the use of the hydrometer, is the treatment of *portland* cement, whereas the intent of the paper is to describe a method of treatment for *portland-puzzolan* cements.

The discussions do not take issue with the authors' statement that turbidimeter methods are not directly applicable to portland-puzzolan cements, which may contain highly translucent puzzolans. It may also be reasonably assumed that the finer particles of translucent puzzolans will present considerable obstacles to microscopic analysis by the ordinary methods.

Further, this paper treats primarily of cements of normal fineness, which constitute the great majority of the cement output, whereas the objections to assumptions regarding the finer sizes are most pertinent for unusually fine cements. Numerous tests made since the presentation of this paper indicate that with increasing fineness of portland cement the specific surface by hydrometer is increasingly lower than the specific surface by turbidimeter. This fact can be accounted for as being due, for the hydrometer method, partly to error in assumption of the average size of the smallest fraction but principally to flocculation of the extremely fine particles over long periods of sedimentation. Tests on fractions of portland cements—0 to 7, 7 to 22 μ, etc.—indicate that above 5 μ there is a reasonable correlation between the weight distributions determined by microscope and by sedimentation, and a close relation between the corresponding surface areas. However, below 5 μ, a combination of various causes results in a doubtful hydrometer analysis for cement of high fineness (specific surface greater than perhaps 2000 sq. cm. per g.).

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[7] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

[8] Research Engineer, Engineering Materials Laboratory, University of California, Berkeley, Calif.

If any reasonable convention be adopted for particle sizes below 10 μ, it is possible to determine a distribution curve for the cement by means of three hydrometer readings within a one-hour period of sedimentation, from which can be calculated a single-value fineness constant. From a purely relative standpoint, in a plant where a given cement and milling conditions are held relatively constant, such an evaluation of the fineness of the portland-puzzolan cement makes possible a rapid method of mill control. With the hydrometer fineness available, regardless of the basis of calculation, the turbidimeter may be adapted to control the mills to the particular fineness required. Such a method is in practical use at several cement plants; and while there may be doubt as to the absolute values of specific surface, yet the product is uniformly controlled to any desired fineness.

The intent of the paper is to describe a rapid and simple method for acceptance testing and for routine control in the grinding of portland-puzzolan cements; and while it is desirable to obtain values near those which may be observed by microscopic analysis, it is not necessary that these values be the same. The authors agree that the hydrometer is not suitable for determination of absolute specific surface on a strictly scientific basis; however, the same statement is true of every other present method with the possible exception of the microscope. The determination of absolute specific surface, however, is merely of academic interest from the standpoint of manufacture and use of cement under present-day conditions.

PETROGRAPHIC STUDIES OF HYDRATED CEMENTS

By L. S. Brown[1] and R. W. Carlson[2]

Synopsis

A method of making distinct thin sections of hardened cement and concrete is described. Micrographs of sections representing various hydrated specimens are presented and discussed. Included among the specimens are (1) normal cement in two finenesses and two water-cement ratios, (2) steam-cured cements, both plain and containing 40 per cent ground quartz sand, (3) portland-puzzolan cements, (4) pure silicates of cement, and (5) coarse fractions of cement. Differences in texture are apparent in most cases.

A few investigations into the nature of the gel in hydrated cement are described and the general structure of the gel is suggested. The importance of the gel, as opposed to calcium hydroxide, in the development of strength, is emphasized. For example, micrographs show an abundance of calcium hydroxide in tricalcium silicate, almost completely hydrated, as compared with substantially no calcium hydroxide in dicalcium silicate, hydrated only fractionally, although both exhibit about equal strength at the age of examination.

The present paper is based on research conducted at the Massachusetts Institute of Technology and made possible largely by a grant from the National Research Council. This paper marks a step in the research program at the Institute on the nature and properties of hydrated cement and its constituents. It is intended to continue and to include studies of dimensional changes of the gel in the program for the coming year.

Introduction

It has been fairly well established that the hydration of portland cement results largely in two substances, crystalline calcium hydroxide and an amorphous hydrous material conveniently termed "gel." These two substances are produced in the hydration of dicalcium silicate and tricalcium silicate, which together make up about 75 per cent of an average portland cement. Recently it has been shown that these two silicates are the only constituents of portland cement that are capable, on hydration, of developing strengths that are comparable with those developed by portland cement, and it has been shown also that tricalcium silicate alone may hydrate, set, and harden like normal portland cement.

From the above considerations it may be seen that the fundamental properties of portland cement, such as hydraulic set, tendency to volume change, porosity, etc., must be largely explained by the individual properties and interrelationships of calcium hydroxide and gel as they are developed in the hydration of the cement. It is not to be inferred that the ferric oxide and alumina compounds are of no moment. Their influence is regarded, in this paper, as a modification of the characters or processes of hydration and set for which the calcium silicates seem fundamentally responsible.

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[1] Research Petrographer, Lone Star Cement Co., Hudson, N. Y.; formerly Research Associate at the Massachusetts Institute of Technology.

[2] Associate Professor of Civil Engineering, Engineering Materials Laboratory, University of California, Berkeley, Calif.

In the present paper there are offered some observations on the internal structure of hydrated and set cements as revealed through microscopic examination. There will first be described briefly the technique of preparation of thin sections for examination, in which the difficulties encountered are themselves informative. There will then be illustrated the structures of hydrated specimens of standard commercial cements, cements with admixtures, and pure calcium silicates of portland cement.

TECHNIQUE OF PREPARATION

The preparation of thin sections of hardened cement pastes for microscopic examination presents unusual difficulties, because the pastes are finely porous and contain an abundance of adsorbed water. The momentary heating required to cement the specimen to the glass slide releases numerous bubbles of vapor, preventing the formation of a good bond. Without a good bond it is difficult to grind the section to the extreme thinness required by the fine-grained character of the cement. Finally, even if a section of satisfactory thinness is obtained, it is almost impenetrable to transmitted light, and by reflected light it is a dense creamy white, although the component mineral substances individually are clear and colorless. These light effects also are due to the adsorbed water abundantly distributed through the hydrated cement in minute pores, the water having a low index of refraction compared with the indices of the other substances.

Removal of the adsorbed water in set cement and impregnation by a resin of higher index of refraction makes it possible to obtain satisfactory thin sections. This procedure not only renders the section itself clear, but also it permits the formation of a good bond with the glass, and permits grinding to exceptional thinness. Removal of the water requires drying from 6 to 15 hr. at 120 to 135 C. Impregnation with resin is accomplished by soaking the face to be sectioned in the melted resin on a hot plate for about $\frac{1}{2}$ hr. Canada balsam can be used, but the synthetic resin "Kollolith" has been found to be more satisfactory. Its index of refraction (1.5354) is about the same as that of balsam and is close to those of both calcium hydroxide and gel; it has an appreciably lower surface tension than balsam, permitting easier penetration, and it does not harden as

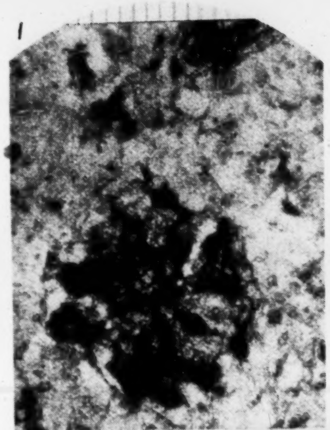
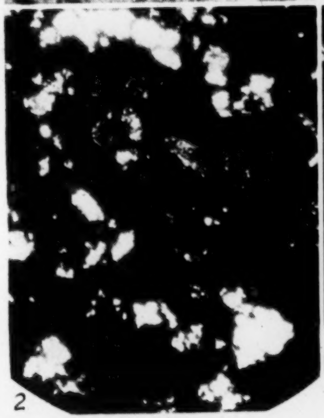
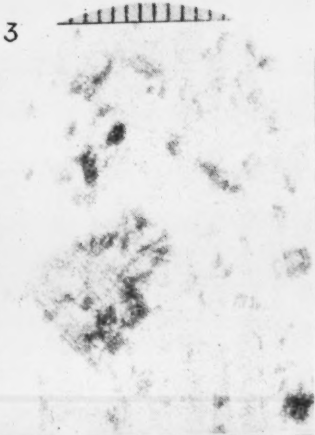
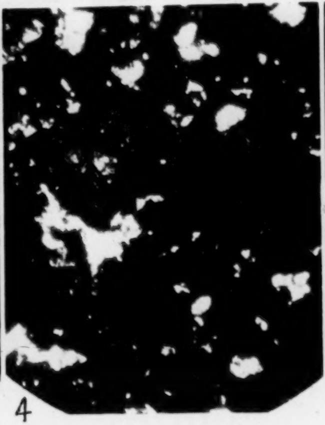
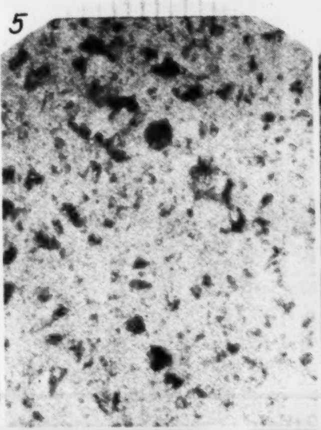
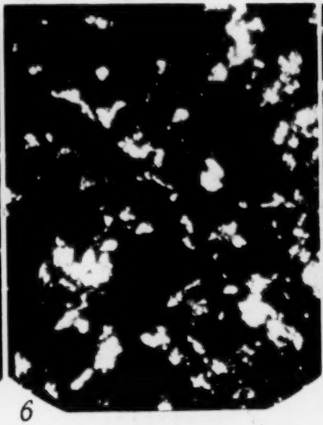
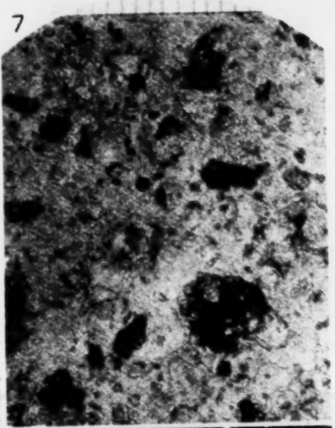

FIG. 1.—Cement of Normal Fineness, $w/c = 1.00$.
FIG. 2.—Cement of Normal Fineness, $w/c = 1.00$ (Crossed Nicols).
FIG. 3.—Cement of Normal Fineness, $w/c = 0.80$.
FIG. 4.—Cement of Normal Fineness, $w/c = 0.80$ (Crossed Nicols).

FIG. 5.—Finely Ground Cement, $w/c = 1.00$.
FIG. 6.—Finely Ground Cement, $w/c = 1.00$ (Crossed Nicols).
FIG. 7.—Finely Ground Cement, $w/c = 0.80$.
FIG. 8.—Finely Ground Cement, $w/c = 0.80$ (Crossed Nicols).

rapidly under prolonged heating. Grinding is accomplished in the usual manner, carborundum being used for the coarse grinding and Bausch & Lomb No. 906 corundum for the finish grinding. The sections are ground in water; this procedure is justifiable as the exposed surface is removed faster than hydration proceeds. Sections prepared as just described are remarkably clear and illustrate internal structures in fine detail.

INTERPRETATION OF PHOTOMICROGRAPHS PRESENTED

The photomicrographs (Figs. 1 to 28) are presented in pairs, those taken in ordinary illumination and those with crossed nicols. With ordinary illumination, nothing appears but unhydrated residual grains of cement, grains of admixture in certain cases, and hydration products, while with crossed nicols interest is centered in but two products, gel and crystalline calcium hydroxide. As all of these are fine-grained substances, it is difficult to indicate the specific nature of every detail of the print.

No symbols are employed to indicate gel. With crossed nicols the gel always appears dark; hence, all dark areas are gel except for holes, which are indicated by the letter "o" in the prints, and crystalline minerals at extinction, which are minimized by rotation of the stage in taking the photograph. Crystalline calcium hydroxide usually is distinguishable by its feathery or indefinite outline and birefringence, and in the occasional cases where the birefringence of admixture minerals tends to cause confusion the letter "h" is placed beside the hydroxide.

DISCUSSION OF THIN SECTIONS

Hydrated Commercial Cement:

On mixing portland cement with water in the proportions commonly employed, it was observed that gel and calcium hydroxide began to form almost immediately. The hydroxide crystallized at numerous centers within the mass, the crystals gradually growing in size as hydration progressed. The gel developed around every cement particle and did not form in isolated units as did the hydroxide. After a few days of hydration, all of the intergranular spaces, excepting the air bubbles, appeared to be filled with either gel or calcium hydroxide.

Four neat specimens were made from portland cement of average chemical composition, ground to two finenesses and mixed in two water-cement ratios. The specific surfaces were 1450 sq. cm. per gram (normal) and 2250 sq. cm. per gram (fine). The water-cement ratios were 0.80 and 1.00 by weight. After the specimens were stored for 3 months at room temperature, thin sections were prepared. Much of the mixing water had been absorbed at this age and, with these high water contents, the cements were well cured.

Figures 1 to 8 are photomicrographs of the thin sections of these four specimens. The first photograph of each section was taken with ordinary light to show the extent of hydration, and the second was taken with crossed nicols to bring out the crystalline structure. The scale of magnification is shown at the end of each photograph, each division representing 0.01 mm. (10 μ). A discrepancy will be noted between magnifications for identical microscopic fields with and without crossed nicols, amounting to approximately 8 per cent reduction in linear magnification with the double polarized light. Since the scale could not be photographed against a dark field, the scales of magnification in prints with crossed nicols is not strictly accurate.

With ordinary light (Figs. 1, 3, 5 and 7), the smaller residual unhydrated grains appear dark and the larger grains have dark borders owing to their high refractive index. Large unhydrated grains remain in the coarse cement, as shown in Figs. 1 and 3; these contain not only dicalcium silicate but also tricalcium silicate, as evidenced by interference color noted in the microscope for corresponding areas of Figs. 2 and 4. Thus, even though tricalcium silicate is known to hydrate rapidly, it appears to be unavailable to ready hydration in the coarse grains.

The figures with crossed nicols reveal significant differences in texture. The contrast is particularly striking between the coarser cement with high water content and the finer cement with low water content (Figs. 2 and 8). Crystals of calcium hydroxide in the former specimen are large and few, whereas in the latter they are much smaller and more numerous. Tracing the changes through the series, a systematic trend may be observed, finer cement and less mixing water resulting in smaller but more numerous calcium hydroxide crystals. Coarser gel structure also was found in the specimens of coarser cement and of higher water content. This was evidenced by the ease with which these specimens soaked up the resin used for impregnation.

Finer grinding and lower water-cement ratios tend to produce a more intimately continuous gel, which is represented by the black areas in Figs. 2, 4, 6, and 8. The black areas also include some hydroxide crystals at extinction; but, in photographing, the stage was oriented so as to reduce these to a minimum. Strength data were not available for these particular specimens, but it may be remarked that finer cements and lower water-cement ratios also tend to produce progressively higher strengths.

Textural Effects of Steam Curing:

Two briquets of steam-cured cements were available for the preparation of sections, both briquets having been molded and allowed to set for 24 hr., and then steamed in an autoclave at 170 C. for 24 hr. One of these contained white portland cement and the other contained identical cement mixed with 40 per cent of quartz sand which had been ground to pass a No. 200 sieve. Both specimens had been made in the standard method for making neat briquets, considering the ground quartz as part of the cement in the second case. Tensile strengths obtained after 24 hr. of steaming were 420 lb. per sq. in. for the plain cement and 980 lb. per sq. in. for the specimen containing ground quartz.[3] The great difference in strength made this an attractive case for thin-section investigation. Thin sections were prepared from the broken briquets with difficulty, because the density was such as to prevent ready penetration of resin.

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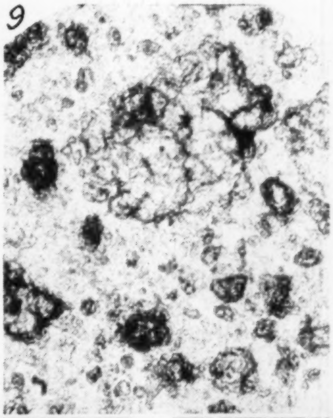

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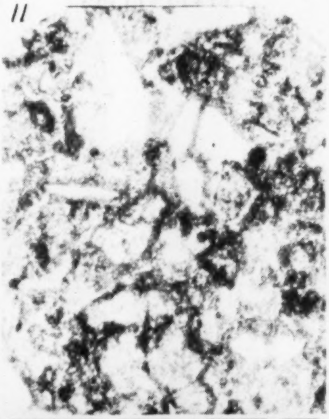

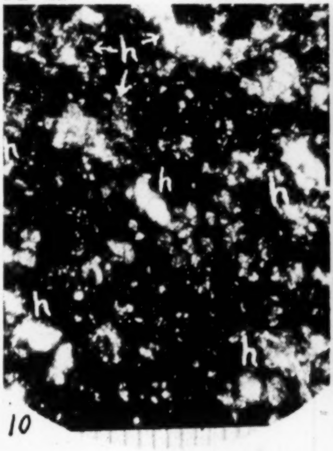

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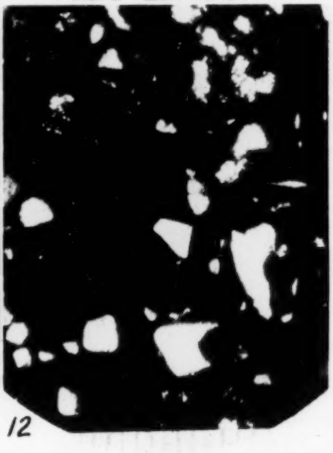
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FIG. 9.—Steam-Cured Neat Cement.
FIG. 10.—Steam-Cured Neat Cement (Crossed Nicols).
FIG. 11.—Steam-Cured Cement Containing 40 per cent Ground Quartz.
FIG. 12.—Steam-Cured Cement Containing 40 per cent Ground Quartz (Crossed Nicols).

Thin sections of the steam-cured briquets as they appear under ordinary illumination are shown in Figs. 9 and 11 for the plain and quartz-admixture specimens, respectively. The grains with dark borders are unhydrated residual cement particles, and it may be observed that the specimens are only slightly hydrated. In view of the slight degree of hydration, the extraordinarily high strengths are remarkable. The unhydrated particles are principally dicalcium silicate, but important amounts of tricalcium silicate also may be observed, the latter appearing under crossed nicols with low interference colors. The angular white areas in Fig. 11 represent quartz grains.

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[3] For other test results on similar steam-cured specimens, see paper by C. A. Menzel, "Strength and Volume Change of Steam-Cured Portland Cement Mortar and Concrete," *Journal*, Am. Concrete Inst., Vol. 6, pp. 125–148 (1934).

Between crossed nicols the two sections exhibit a marked difference (see Figs. 10 and 12). In this case, the crystalline areas appear light. In Fig. 10, the unhydrated cement grains may be detected, with some difficulty, from their corresponding positions in Fig. 9. The highly irregular inter-granular crystals with feathery borders are calcium hydroxide. In their size and distribution the hydroxide crystals resemble those of a "normal" cement (Figs. 2 and 4), except that many of them occur as unusually small crystals distributed through the mass.

All of the crystalline areas except those of the unhydrated grains appeared to be calcium hydroxide, since they exhibited substantially the same birefringence, and all determinable interference figures indicated uniaxial negative character, as of calcium hydroxide. No crystals were observed corresponding to Thorvaldson's recently described crystalline calcium silicate hydrate,[4] but this is not surprising because Thorvaldson found them to develop with some difficulty in white cement. The gel mass, seen more distinctly in the microscope than is possible in the photograph, gave the appearance of having an abundance of extremely fine crystals of calcium hydroxide distributed through it. The gel was apparently more dense and brittle, however, than the gel in hydrated cement not steam-cured.

In contrast, the specimen containing ground quartz, likewise steam cured, reveals very little crystalline material other than the quartz and residual cement grains (see Fig. 12). The gel mass appears almost completely isotropic, containing only a few minute birefringent spots (probably calcium hydroxide), always immediately adjoining unhydrated tricalcium silicate. Since almost no calcium hydroxide is present, it is indicated that this hydration product has combined with the quartz. Judging from the small quantity of quartz grains present, it is also indicated that the smaller grains of quartz have been consumed. It has long been known that silica and calcium hydroxide will react at moderate temperatures in the presence of moisture, as demonstrated clearly by Thorvaldson.[4] The surfaces of the surviving quartz grains, most of which are comparatively large, show in this case a very delicate etching, indicating an attack by the calcium hydroxide that is consistent with the unusually abundant gel development. Again,

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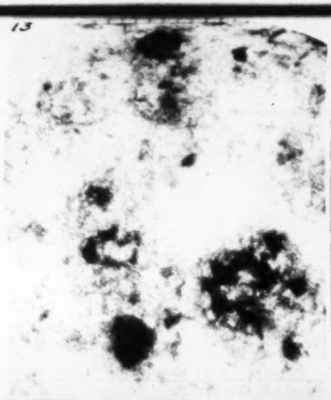
[4] T. Thorvaldson and G. R. Shelton, "Steam Curing of Portland Cement Mortars," *Canadian Journal of Research*, Vol. 1, pp. 148–154 (1929).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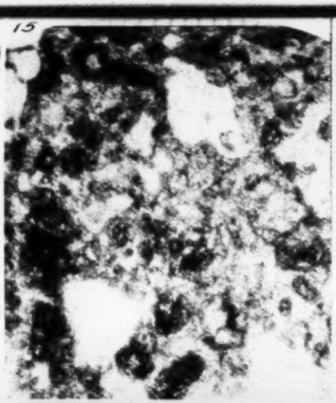
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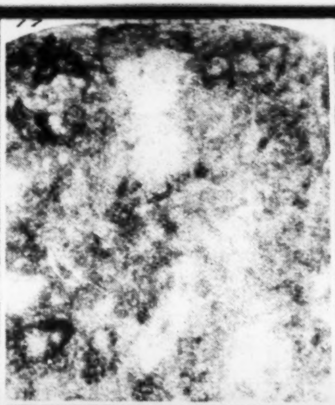
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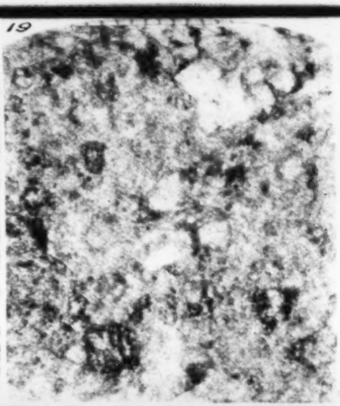
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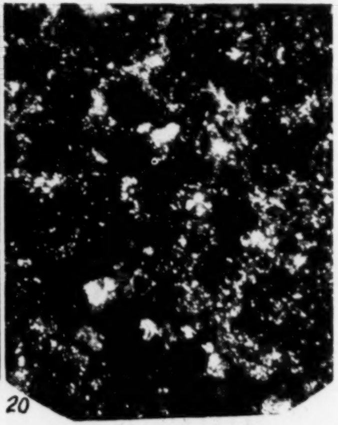
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FIG. 13.—Portland-Puzzolan Cement Containing 45 per cent Ground Granite.

FIG. 14.—Portland-Puzzolan Cement Containing 45 per cent Ground Granite (Crossed Nicols).

FIG. 15.—Portland-Puzzolan Cement Containing 20 per cent Volcanic Tuff.

FIG. 16.—Portland-Puzzolan Cement Containing 20 per cent Volcanic Tuff (Crossed Nicols).

FIG. 17.—Portland-Puzzolan Cement Containing 25 per cent Diatomaceous Shale.

FIG. 18.—Portland-Puzzolan Cement Containing 25 per cent Diatomaceous Shale (Crossed Nicols).

FIG. 19.—Adulterated Portland Cement Containing 20 per cent Limestone.

FIG. 20.—Adulterated Portland Cement Containing 20 per cent Limestone (Crossed Nicols).

there is no evidence of crystallization of any of the gel. It is interesting to recall while noting the abundant gel development in this specimen that the briquet from which it was made exhibited the remarkable tensile strength of 980 lb. per sq. in. Since in this specimen substantially no calcium hydroxide can be observed, it is indicated that the gel is the main cementing material.

Puzzolanic and Other Admixtures:

Four admixture specimens were submitted for examination by W. C. Hanna, Chief Chemist of the California Portland Cement Co. These were neat pats containing portland-puzzolan cements and prepared according to the standard method for soundness tests. The portland-puzzolan cements were made by intergrinding a high-lime clinker with (1) 45 per cent of crushed granite, (2) 20 per cent of volcanic tuff, (3) 25 per cent of diatomaceous shale, or (4) 20 per cent of crushed limestone. The pats were stored in damp air for 24 hr. after preparation, and then in water at 70 F. for approximately one year.

These specimens are illustrated in thin section in Figs. 13 to 20. With ordinary illumination, an important amount of unhydrated cement is observed in every case. While the unhydrated particles usually are of dicalcium silicate, tricalcium silicate is again present in every case. The specimen containing crushed granite is evidently more thoroughly hydrated than any of the others. It is interesting to note that both the amount of water per unit of cement and the amount of pore space in which the hydration products of the cement might form were greatest in the case of the granite admixture, possibly accounting for the extensive hydration. Auxiliary tests showed the granite to be almost inert with lime at ordinary temperatures.

The textures of the hydrated structures show considerable dissimilarity. With crushed granite (Fig. 14) the texture is that of coarse cement; the hydroxide appears in large areas which are not always single crystals and thus not clear-cut in the photograph. Volcanic tuff (Fig. 16) has been somewhat more effective; in this case the hydroxide areas are numerous but considerably smaller. Although separate tests have shown the tuff to be fairly active with lime, it is apparent that the 20 per cent tuff used in this case was insufficient to absorb much of the hydroxide, at least up to this age. Figure 18, illustrating the cement interground with diatomaceous shale, is notably different. The gel is abundant and is essentially continuous, being interrupted only by occasional small crystals of calcium hydroxide. It is likely that the shale has absorbed much of the hydroxide, separate tests showing the shale to be very active with lime. The effects of crushed limestone interground with cement (Fig. 20) are difficult to evaluate. The limestone particles are so numerous and possess such high birefringence that they dominate the field. Direct observation of the microscopic field shows a fair amount of calcium hydroxide, but the total amount and crystal size seem to be far smaller than in a normal cement or in the cement containing crushed granite. The reason for this is not clear. Hydration appears to be less extensive, as evidenced by the number and size of unhydrated grains, but it seems improbable that the difference is sufficient to account for lack of the hydroxide. While the disparity may be entirely in the dominance of the primary carbonate particles, the effect of the calcium carbonate seems to offer a promising subject for further study.

A consistent correlation was noted between the character of the observed micro-structures and the tensile strengths developed by corresponding specimens of standard mortar. Strengths shown by the various admixtures, from highest to lowest, were in the following order: diatomaceous shale, tuff, limestone, and granite. The 3-month strength shown by the granite was less than half of that shown by the shale, and a comparison of the corresponding photographs shows a much more continuous hydrated structure in the case of the shale, with calcium hydroxide more evenly distributed and less in total amount. Here, again, the specimen with the less calcium hydroxide developed the higher strength.

The combined volume of gel and calcium hydroxide apparently was sufficient to fill even the large amount of void space existing in the granite specimen, in which there was only 55 per cent of portland cement. Since the gel seems to fill the intergranular voids completely, the gel in the granite specimen logically should be more porous than that in a specimen having a higher cement content and a more active puzzolanic material, a system with potentially more gel for a similar ultimate external volume. The gel thus seems to possess the remarkable property of adjusting its density, within limits, to conform to the space available for its development. It was noted, during the process of impregnating the specimens for the preparation of thin sections, that the granite specimen was the more porous, absorbing the resin easily and quickly.

Tricalcium Silicate (C_3S) *and Dicalcium Silicate* (C_2S):

Two specimens of hydrated C_3S and C_2S, as pure compounds, were available for study. These were in the form of small cylinders, about $1\frac{1}{2}$ by $\frac{3}{4}$ in. in size, originally prepared and used for water-absorption studies. They were prepared by pulverizing each of the calcium silicates, mixing each with water to a slurry, placing each in a thimble, immersing in water, and keeping for $1\frac{1}{2}$ yr. before sectioning.

Figure 21 is a section of the tricalcium silicate with ordinary illumination, showing hydration to be practically complete; only one large un-

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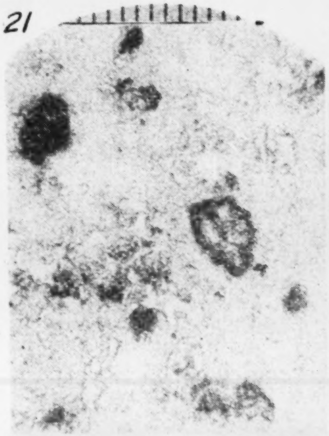

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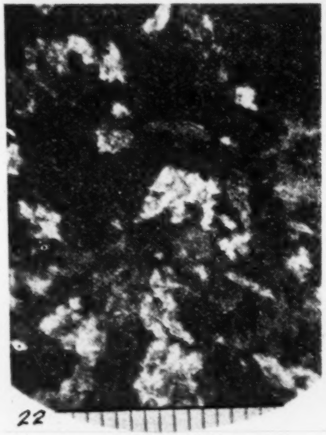

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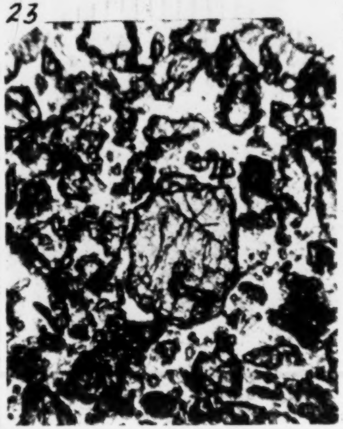

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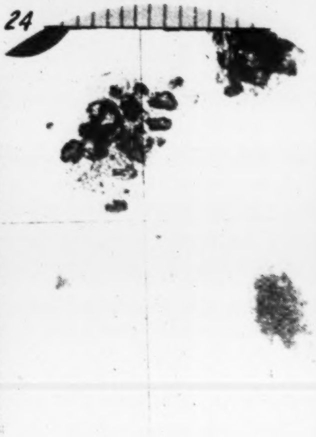

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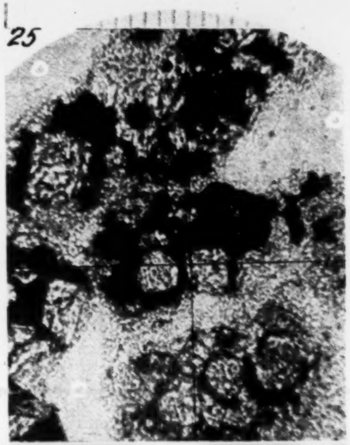

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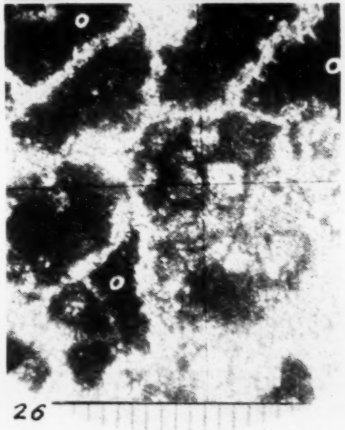


FIG. 21.—Pure C_3S Hydrated 1½ yr.
FIG. 22.—Pure C_3S Hydrated 1½ yr. (Crossed Nicols).
FIG. 23.—Pure C_2S Hydrated 1½ yr.

FIG. 24.—Hydrated C_2S Grains in Index Oil.
FIG. 25.—Coarse Fraction of Cement, Hydrated 4 months.
FIG. 26.—Coarse Fraction of Cement, Hydrated 4 months (Crossed Nicols).

hydrated grain appears in the photograph. Figure 22, representing the same field between crossed nicols, reveals a structure similar to that of a hydrated portland cement, although the hydroxide crystals are larger, more numerous, and better developed. In this photograph is a confirmation of a previous statement that tricalcium silicate by itself will set and harden in a manner similar to that of normal cement. A corresponding specimen had a compressive strength of about 10,000 lb. per sq. in., 60 per cent of which was reached in 7 days. The dicalcium silicate, shown with ordinary illumination in Fig. 23, is far from complete hydration; the unhydrated grains are large, clear, and regular in outline. In spite of the low degree of hydration, companion specimens also gave a compressive strength of 10,000 lb. per sq. in. at the age of $1\frac{1}{2}$ yr., the strength at 7 days being negligible. No photograph is shown with crossed nicols, as the field, with the exception of the unhydrated grains, is completely dark, indicating that all hydrated material is gel. Thus the strength-producing property of the gel is again confirmed. In studying the thin section, but one crystal of hydroxide could be found; this was small and might well be attributed to free lime in the initial material.

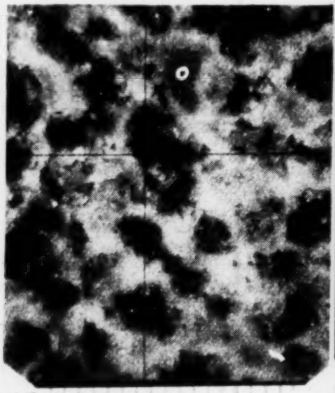
POROSITY OF HYDRATED CEMENT

The permeability (porosity) of hydrated and set portland cement and concrete is a well-known character. It has been indicated in preceding paragraphs that the porosity may be a property of gel structure rather than of intergranular voids. Two experiments were performed for the purpose of yielding more definite information on this question.

The first experiment consisted in the addition of a deep crimson dye, soluble in resin but not in water, to the resin used for impregnating the specimen. This was done in an effort to make more clearly visible in the thin section the channels or pores into which the resin penetrated. The experiment was not wholly successful, in that the color, though almost opaque through $\frac{1}{8}$ in. thickness in the resin, became very faint through the thickness of the section (20 μ). The best illustration was obtained with the hydrated pure C_3S previously described, shown with plane transmitted light in Fig. 21, where the tint shows even less plainly than could be observed directly in the microscopic field. However, it may be noted that the areas of calcium hydroxide crystallization are white (colorless) whereas all of the gel area is uniformly tinted. No separate individual channels could be distinguished, even at 1000 diameters magnification, whence it may be concluded that the pores are exceedingly numerous, but of submicroscopic dimensions.

The objective of the second experiment was to make intergranular pore space more distinctly visible. To this end neat cement pats were prepared from the coarser fractions of an air-classified cement supplied through the courtesy of F. R. McMillan, Director of Research of the Portland Cement Association. Two fraction sizes were employed separately, 45 to 60 μ and 60 to 85 μ. Pats were prepared by mixing to a stiff consistency with water, then storing in moist air until used. The first sections were prepared at the age of 4 months, when the pats had set quite hard.

Figure 25 shows a section of a pat made from the 60 to 85 μ particles under ordinary illumination. The original grains of cement are clearly visible, though being themselves largely aggregates of crystals of dicalcium and tricalcium silicates bound together with dark interstitial material. Much of the intergranular space has been filled with gel and calcium hydroxide, even though hydration is not extensive and intergranular spaces are large with these coarse particles. The intergranular areas that yet remain open are marked with the letter "o." However, these areas do not have the regularity or characteristic shape of original inter-particle spaces. The gel does not seem to have developed evenly around each particle to form the hydrated shells or uniform cloaks of gel, as sometimes pictured. Instead, the gel seems to have grown abundantly into those spaces offering the greatest freedom for growth, especially when such spaces are adjacent to C_3S minerals. With crossed nicols (Fig. 26) the gel appears completely birefringent (white in the photograph instead of dark), due to the formation of fine-grained calcium carbonate distributed through the gel, the transformation evidently having occurred readily in the coarsely porous gel structure formed from the coarse particles. The manner in which an apparently weak gel has completely filled many of the inter-particle spaces is of especial interest; it appears that the gel has filled some spaces that are as much as 100 μ in width.



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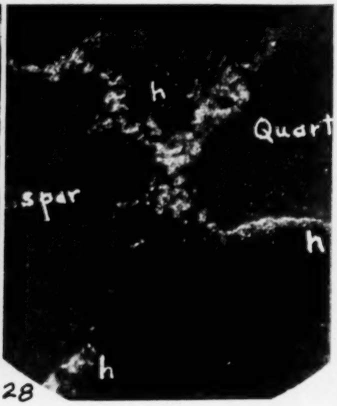

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FIG. 27.—Coarse Fraction of Cement, Hydrated 4 months (Reflected Light).
FIG. 28.—Concrete Specimen with Characteristic Calcium Hydroxide Crystals (Crossed Nicols).

A section of a pat made from particles of the size 45 to 60 μ as it appears by reflected light is shown in Fig. 27. The section was prepared after 8 months of storage of the pat in moist air. Not all of the moisture was removed in preparation of the section, and the photograph was taken with reflected light only. Here again the initial particles (dark) are clearly visible, again as mineral clusters. Intergranular space is filled, with the exception of a few small openings, marked with the letter "o" in the figure. The gel seems to have filled most of the available space, and again no separate cloaks of gel are visible. The gel exhibits the brilliant creamy whiteness that is characteristic of hydrated portland cement under the microscope, especially when the cement is not thoroughly dried. Although this section is ground to the standard thickness (0.03 mm.), yet with transmitted light (not shown) the field is nearly dark. These observations again indicate a minute porosity; the pores are at least partially filled with water, the refractive index of which is markedly lower than that of the gel.

Observations recorded in the preceding paragraphs lead to the conclusion that the porosity of a well-mixed and well-cured portland cement or concrete is largely, if not principally, a function of gel structure. In the experiment with dyed resin no separate pores or channels could be discovered, while gel areas were definitely and uniformly tinted. The experiment with coarse fractions illustrated the ease with which the developing gel completely filled comparatively enormous intergranular voids. The use of the usual cement, with continuously graded sizes, greatly decreases the average dimension of intergranular pore space, spaces that can be easily filled in a comparatively short time.

CHEMICAL AND PHYSICAL NATURE OF GEL IN PORTLAND CEMENT

R. H. Bogue and his co-workers,[5] and others, have shown that the gel of portland cement is derived mainly from hydration of the dicalcium and tricalcium silicates. Studying the hydrated pure compounds, they have observed that the gel chemically is a substance of variable water content, containing at equilibrium under ordinary temperatures and water contents lime and silica in an empirical ratio approaching that of $2CaO \cdot SiO_2 \cdot xH_2O$. Divergence from this ratio, however, is sufficient to indicate more or less clearly that the ratio of lime to silica in the gel is not stoichiometric.

It has been demonstrated in preceding paragraphs that the gel is a finely, but highly, porous medium. In this respect it is similar to silica gel and many other inorganic gels. Thus it may be properly classified as a member of the group known as "irreversible" or "rigid" gels, which are characterized by a firm but open structure with much internal space of capillary dimensions.

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[5] R. H. Bogue and William Lerch, "Hydration of Portland Cement Compounds," *Paper No. 27*, Portland Cement Assn. Fellowship, National Bureau of Standards, August, 1934.

On the other hand, the apparent lack of stoichiometric precision in its constitutional formula supports the conclusion that the gel should more properly be considered to be silica gel with adsorbed calcium and oxygen atoms. Certain tests and observations bearing on this possibility are summarized below:

1. *Chemical Formula of the Gel.*—Pure tricalcium and dicalcium silicates were powdered to pass completely through a No. 200 sieve. Portions of each were gaged with 75 per cent, by weight, of water, and sealed in rubber balloons, the specimens thereafter being kneaded frequently within the container, removed after 10 days and dried in a desiccator. After drying 3 days, duplicate portions were removed and titrated for free lime (as calcium hydroxide), according to Bogue's modification of Emley's method. The tricalcium silicate titrated 19.2 per cent $Ca(OH)_2$, essentially the same value (19.3 per cent) found by Bogue at 6 months. Thus the formula of the gel again was found to be nearly disilicate plus water. The dicalcium silicate yielded no $Ca(OH)_2$, although it must be pointed out that microscopic observation disclosed hydration in this case to be essentially negligible, whereas hydration of the trisilicate appeared to be complete.

2. *Refractive Index Observations.*—The dried gel from the tricalcium silicate exhibited an index of refraction close to 1.515. Precise measurements are difficult to obtain, due to the prompt exudation of fine water globules upon placing the powder in the refractive index oil. Heating in a muffle furnace gradually increased the refractive index to a maximum of 1.695 after 1 hr. at 900 C.

A similar behavior was found in a separate portion of hydrated dicalcium silicate submitted by Mr. Bogue. This was a powder specimen that had been hydrated about 1½ yr., with many cycles of drying, regrinding, and remixing with water to facilitate hydration. The specimen contained an abundance of gel, but no appreciable calcium hydroxide. Refractive index of the air-dried powder was 1.530; heating for 1 hr. at 900 C. raised the index to 1.695.

These values are not well in accord with those for silica gel, which attains an index close to 1.470 with similar heat treatment. It is possible, however, that adsorption of calcium and oxygen atoms might raise the the refractive index of silica gel to the values observed.

The difficult hydration of dicalcium silicate is illustrated by the observation that, even with repeated grinding and remixing with water, this specimen was hardly more than 25 or 30 per cent hydrated. Some of the grains are shown in Fig. 24, mounted in an oil of refractive index 1.535. The outlines of the gel granules may be clearly discerned. Within the mass the residual particles of unhydrated disilicate stand out boldly because of a much higher index (1.730).

3. The gel of cement, like silica gel, does not appear to be a true

hydrate. At ordinary temperatures either of these gels may hold a fair amount of adsorbed water, but the amount is variable, being high when the pore structure is fine and the humidity is high, and being low when the temperature is high.[6] When the dissociation temperature of calcium hydroxide is approached, practically no water remains in the gel. This fact was verified for completely hydrated C_3S by noting that practically all of the loss in weight between the temperatures of 350 and 550 C. could be accounted for by the dissociation of calcium hydroxide that occurs between these temperatures. Thus, water is not a necessary part of cement gel and it has long been known that it is not a necessary part of silica gel.

4. The gel in all stages of dryness apparently is completely amorphous; no evidence of fine crystallinity can be perceived although much of the CaO, which might have been expected to be in the crystalline hydroxide form, can be shown to be within the gel. The gel in the moist form reacts instantly with CO_2, filling the gel with minute $CaCO_3$ crystals. Many evidences of this reaction were observed and Fig. 26, previously discussed, has illustrated the complete carbonation of the porous gel produced by the hydration of coarse particles of cement. The carbonation proceeds more easily in the gel than in areas of recognized crystalline hydroxide, although the gel after thorough drying at high temperature apparently is unaffected by dry carbon dioxide. This behavior may indicate that the calcium oxide is only a stable part of the gel when in an alkaline medium, because the action of carbonic acid is typical of that of other weak acids. The probable result of the reaction of acids with cement gel is to convert it into silica gel.

5. Hydrochloric acid, even when very dilute, will accordingly react with the lime in cement gel without destroying the gel, again probably leaving silica gel. Furthermore, Thorvaldson has shown[4] that crystalline calcium hydrosilicate is decomposed by hydrochloric acid but that a skeleton is left (probably SiO_2). Thus, whether in gel form or in crystalline form, the fundamental structure of hydrated calcium silicate seems to be an open SiO_2 frame. The spaces within the frame can apparently cover a wide range of dimensions, from molecular sizes up to perhaps 1000 times this size, depending upon the conditions of formation. The crystalline calcium hydrosilicate may possibly represent a particular form of the "gel," when the pores are of molecular dimensions and are more or less filled with definite amounts of CaO and H_2O.

SYNERESIS

All gels, rigid and non-rigid, exhibit more or less strikingly the phenomenon known as "syneresis," that is, a tendency to shrink of themselves, with expulsion of free liquid. In this regard, silica gel is no exception, and it might be expected that portland-cement gel would exhibit the same behavior. Some evidence of such behavior may be found in Fig. 28, which is a section of concrete photographed with crossed nicols. Portions of two large pieces of aggregate, feldspar and quartz, may be seen outlined by clear areas of calcium hydroxide crystallization, with the gel (dark) occupying the remainder of the field. Crystallization of the hydroxide in these comparatively large areas evidently indicates a migration of the hydroxide solution from the points of generation. The crystals often appear to be larger than the original interparticle spaces could have been. This large size of crystals may be evidence of syneretic behavior, although this conclusion remains to be proved.

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[6] R. Wilson and F. A. Martin, "Water Retained in Hardened Cement Pastes," *Journal*, Am. Concrete Inst. Vol. 6, January–February, 1935, pp. 272–279.

No evidence has been found to prove that calcium hydroxide crystallizes within the pores of the gel structure. Figure 28 is an illustration of the development of calcium hydroxide adjacent to the gel. Migration of the solutions definitely must have occurred. As no visible channels have been observed, it may be concluded that the gel structure itself, by means of its submicroscopic pores, has provided the channels of migration; and in this respect syneresis seems definitely to occur. That the calcium hydroxide does not crystallize in the pores of the gel is further proved by the fact that the pores are always found to be open and permeable.

SUMMARY

As observed by microscopic examination of thin sections, set cement consists mainly of three components: calcium hydroxide crystals, calcium silicate gel, and unhydrated cores of cement grains. The gel, which is porous, appears to fill substantially all intergranular spaces not filled by the hydroxide. Thus the degree of porosity of the gel varies with the conditions under which it is developed. Coarse cement and high water content produce more porous gel than do fine cement and low water content. Correspondingly, coarse cement and high water content produce large crystals of calcium hydroxide and low strength.

The strength of portland cement is probably developed by the gel acting as a void-filling glue, rather than by any type of crystallization. Other things being equal, the less calcium hydroxide observed in a thin section, the greater the corresponding strength.

Because with a given water-cement ratio and a given mix the intergranular pore space is not greatly altered in total amount by changing the nature of the cement, and because substantially all of the pore space is found to be filled either with gel or with calcium hydroxide after cement hydration, that cement which hydrates most completely should produce the densest gel and the strongest specimen. This observation seems to be in line with the facts that greater strengths are shown by (1) finer cements,

(2) greater ages of hydration, (3) replacement of dicalcium silicate by tricalcium silicate (for early ages), and (4) higher curing temperatures.

Steam curing, after brief preliminary storage at normal temperature, is found to produce substantially the same hydration products as are produced by prolonged curing at normal temperature, except that the gel is noticeably more dense and perhaps approaches crystallinity. When 40 per cent of silica is added before steam curing, most of the calcium hydroxide disappears, probably through combination with the added silica, and the greater strength observed is attributed to the formation of more gel through this puzzolanic action.

Puzzolanic materials, which contain silica capable of combining with calcium hydroxide in the presence of moisture, appear to reduce the amount of hydroxide and to increase the amount of gel in set cement. The lower hydroxide content in hardened portland-puzzolan cements can be observed qualitatively in thin sections. The lack of hydroxide is most noticeable when the specimens contain very active puzzolanic materials such as diatomaceous shale. Thus, the study of thin sections offers a rough but informative means of determining the effectiveness of puzzolanic materials in absorbing calcium hydroxide from hardened cements.

The ability of a small quantity of very porous gel to fill relatively large intergranular spaces is illustrated in thin sections made from cement containing only coarse particles. In this case the hydration is slight and the intergranular spaces are large, yet a majority of the spaces, even up to 50 or 100 μ in size, are filled with gel. The gel in such a case is shown to be very porous by the rapidity with which it is attacked by carbon dioxide, and by its ready absorption of melted resin.

The moist gel of portland cement is more readily attacked by exposure to carbon dioxide than are the calcium hydroxide crystals. The carbonation of the gel is found to leave a skeleton, supposedly silica gel, with minute calcium carbonate crystals distributed through it. The more coarsely porous the gel, the more readily it is attacked by the carbon dioxide. Dry gel appears to be immune to attack by carbon dioxide.

The gel of portland cement contains normally (1) the oxides of calcium and silica in about the same relative percentages found in dicalcium silicate, and (2) an indefinite amount of water. The higher the temperature, the lower the water content, until at a temperature near the dissociation temperature of calcium hydroxide practically no water remains in the gel. As the dicalcium silicate contains about the same percentage of calcium as does the gel, this silicate does not hydrolyze noticeably under ordinary conditions. Not only by chemical analysis but also by examination of thin sections, the only hydration product of dicalcium silicate is found to be the calcium silicate gel. This gel has tremendous cementing power, producing in a small neat cylinder a compressive strength of 10,000 lb. per sq. in. at

1 yr., even though the hydration at this age is probably less than one-fourth complete.

The gel produced by the hydration of dicalcium silicate is found to be similar to that resulting from the hydration of tricalcium silicate. In either case, a wide range in density and corresponding pore size may be observed, depending upon a number of factors such as temperature, age, water content, etc. At early ages of hydration, the gel of dicalcium silicate is likely to be more porous than the gel of tricalcium silicate.

No evidence is found indicating any crystallization of calcium hydroxide within the gel. The pores of the gel appear to remain open and to serve as channels of migration for the calcium hydroxide, which is liberated from the core of each cement grain only when the core is accessible to water by means of these same channels.

EFFECT OF DEPARTURE FROM PLANENESS OF BEARING SURFACES ON THE COMPRESSIVE STRENGTH OF 2-IN. MORTAR CUBES[1]

By J. R. Dwyer[2]

Synopsis

Study was made of the variations in compressive strength of 2-in. mortar cubes resulting from various departures from planeness in the surfaces of the bearing blocks. Plane, concave and convex bearing surfaces were included and specimens were tested for each of 7 bearing conditions and 4 ages.

The tests were designed to furnish additional data for use in future considerations of certain of the requirements for molds and bearing blocks as now appearing in the A. S. T. M. Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 34 T).[3] The results of the tests indicate that the present requirements for planeness of blocks and molds are not unduly stringent.

Introduction

The investigation described in this paper was prompted by discussions of the Society's Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 34 T)[3] which specifies that the load shall be applied to faces of the cube that were in contact with the mold faces and that no cushioning or bedding material shall be used. Experience has shown that under this method of test the faces of bearing blocks and cubes must closely approach planes if concordant results are to be obtained.

The work of other investigators, particularly that of H. F. Gonnerman,[4] has shown that if the faces of concrete and cement specimens were decidedly non-plane, the observed compressive strengths would be lower. Since the Cement Reference Laboratory inspects and certifies equipment in cement testing laboratories, it appeared very appropriate that the laboratory make this investigation in order to secure additional data to assist Committee C-1 on Cement in future considerations of the Tentative Method C 109 – 34 T.

In this investigation the surfaces of the bearing blocks applied to faces of the cubes were varied. Cubes having bearing faces closely approximating planes were tested between bearing blocks which were (*a*) both plane,

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associate, Cement Reference Laboratory, National Bureau of Standards, Washington, D. C.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 743 (1934); also 1935 Book of A. S. T. M. Tentative Standards, p. 426.

[4] H. F. Gonnerman, "Effect of End Condition of Cylinder in Compression Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 1036 (1924).

(*b*) both concave, and (*c*) both convex. This comparison not only provides data bearing directly on bearing blocks, but it also furnishes information regarding cube mold faces since there are no experimental data showing that there is any difference in strength observed between testing between spherical block surfaces a cube having plane faces and testing between plane blocks a cube having the same curvature of faces as the bearing blocks mentioned in the preceding condition.

SCOPE OF TESTS

Molds.—The molds, 3-gang, were of recent design and were made of cold-rolled steel. The side plates were in contact with the loading faces of the cubes and the rolled surfaces of these sides were true planes well within the tolerance of 0.001 in., the requirement for new molds.

Specimens.—The specimens were all made from one lot of high-early-strength portland cement in the belief that the earlier time tests with this cement would furnish about the same information on the effect of the loading conditions as would tests of an ordinary portland cement tested at greater ages.

The cubes were all prepared in strict accordance with the Tentative Method C 109 - 34 T except: (1) each batch (cement 705 g., sand 1950 g., and water 375 ml.) was larger in order to provide one specimen for each loading condition; (2) a small hard-rubber tamper was used in accordance with the Progress Report of the Working Committee on Plastic Mortar Tests, Committee C-1, December, 1935. On each of 8 days there were made 3 cubes for each loading condition at each of 4 test ages.

All of the specimens were prepared in a room maintained at 70 ± 2 F. and at 60 ± 5 per cent relative humidity. The molds and materials were at room temperature. The temperature of the moist closet in which the cubes were stored for the first 24 hr. was 70 ± 2 F. and the relative humidity was above 95 per cent. The storage tank temperature was nearly the temperature of the room.

A straight edge was applied to the bearing surfaces of each cube just prior to test and it was not considered necessary to rub or grind any of the cubes.

Bearing Blocks and Loading Conditions.—The bearing blocks were steel cylinders, about 3 in. in diameter and 2 in. high. The blocks at top and bottom of the cubes were duplicates. The blocks which did not have plane faces had spherical surfaces, either concave or convex, the surfaces being defined by the height of the mid-ordinate from a plane section of the sphere 2 in. in diameter.

There were 7 loading conditions, as follows:

1. Plane.
2. Concave, mid-ordinate 0.001 in.

3. Concave, mid-ordinate 0.002 in.
4. Concave, mid-ordinate 0.004 in.
5. Convex, mid-ordinate 0.001 in.
6. Convex, mid-ordinate 0.002 in.
7. Convex, mid-ordinate 0.004 in.

Twenty-four cubes were tested under each of the 7 loading conditions and at each of the ages 1, 3, 7 and 28 days—a total of 672 cubes.

Although it was realized that non-plane bearing block faces, and those cube faces which are not planes (and therefore the corresponding surfaces of the molds in which the cubes were made) are not necessarily spherical, it was believed, on the basis of actual observations on such equipment, that the results obtained with spherical bearing surfaces would closely approximate the results obtained with some of the surfaces actually in use and having the same mid-ordinates. The present Tentative Method C 109 – 34 T allows a tolerance not exceeding 0.001 in. departure from a plane for the faces of bearing blocks in use and also for the faces of new molds, and also allows a tolerance not exceeding 0.002 in. for the faces of molds in use. Departures as large as 0.004 in. have been observed in bearing blocks and molds in use.

All of the cubes were tested in the same machine. The load was applied by a hydraulic jack and the magnitude of the load was indicated by a separate hydraulic system. A dial having a capacity of 10,000 lb. was used for the lower loads and a dial having a capacity of 50,000 lb. for the higher loads. The testing machine had been calibrated several months before these tests were made and had been found to comply with the A.S.T.M. Tentative Methods of Verification of Testing Machines (E 4 – 35 T).[5]

METHOD OF TESTING

The cubes were centered between the bearing blocks, with the faces which had been against the side plates of the molds in contact with the bearing blocks. The cube and bearing blocks were carefully centered under the spherical bearing block of the upper head of the machine. The rate of loading, carefully controlled, was the same for all cubes—3000 lb. per sq. in. per min.

RESULTS

The compressive strengths are given in Table I. No results were discarded and no specimens replaced by substitutes.

Concordance of results, as judged by other studies on similar test specimens, appeared quite satisfactory. This was true not only for the daily averages, but also of the agreement between the 3 individual results from which the daily averages were calculated. Among the 224 sets of 3 specimens each, there were only 21 sets for which the mean deviation of the 3 results from their average exceeded 3 per cent and in only 5 of these cases was it in excess of 4 per cent. The grand average of these mean deviations within each set of 3 specimens, calculated for the 8 sets tested for any one loading condition and age, ranged from 1.3 to 2.8 per cent.

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[5] *Proceedings*, Am. Soc. Testing Mats. Vol. 35, Part I, p. 1303 (1935); also 1935 Book of A. S. T. M. Tentative Standards, p. 1427.

Table I.—Compressive Strength of 2-in. Mortar Cubes.
Each value is the average for 24 cubes.

Loading Condition	Bearing Block	Compressive Strength, lb. per. sq. in.			
		Age, 1 day	Age, 3 days	Age, 7 days	Age, 28 days
1......	Plane	1560	3440	4560	5610
2......	Concave, mid-ordinate 0.001 in.[a]	1570	3450	4560	5580
3......	Concave, mid-ordinate 0.002 in.[a]	1580	3480	4570	5560
4......	Concave, mid-ordinate 0.004 in.[a]	1570	3360	4370	5200
5......	Convex, mid-ordinate 0.001 in.[a]	1540	3380	4470	5580
6......	Convex, mid-ordinate 0.002 in.[a]	1520	3310	4430	5460
7......	Convex, mid-ordinate 0.004 in.[a]	1510	3220	4190	5170

[a] Height of mid-ordinate from a plane section 2 in. in diameter.

Table II.—Coefficient of Variation in the Strengths Obtained on Eight Different Days.
Each of the 8 daily strength values is the average for 3 cubes.

Loading Condition	Bearing Block	Ratio of Standard Deviation of the Eight Values to Their Average, per cent			
		Age, 1 day	Age, 3 days	Age, 7 days	Age, 28 days
1......	Plane	1.8	1.5	3.1	2.1
2......	Concave, mid-ordinate 0.001 in.[a]	1.9	0.8	2.8	2.4
3......	Concave, mid-ordinate 0.002 in.[a]	1.0	2.1	2.0	1.7
4......	Concave, mid-ordinate 0.004 in.[a]	1.4	1.1	2.2	2.3
5......	Convex, mid-ordinate 0.001 in.[a]	1.6	1.9	2.2	1.9
6......	Convex, mid-ordinate 0.002 in.[a]	2.1	1.6	2.1	1.9
7......	Convex, mid-ordinate 0.004 in.[a]	2.0	1.8	3.2	2.1

[a] Height of mid-ordinate from a plane section 2 in. in diameter.

A more definite measure of the degree of reproducibility of the tests is furnished by Table II wherein are shown, for each of the bearing conditions and ages, the dispersion of the individual daily averages with relation to their respective grand averages. These measures of dispersion are calculated according to the method outlined in the 1933 A.S.T.M. Manual on Presentation of Data.[6]

The ratios of the strengths obtained with the curved blocks to those obtained at corresponding ages with the plane blocks are so plotted in Fig. 1 that there can be readily followed the variations in the observed compressive strengths due to various combinations of bearing surface and strength of mortar.

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[6] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 453 (1933).

For cubes tested between concave bearing blocks having mid-ordinates not exceeding 0.002 in. (loading conditions 2 and 3) the strengths were nearly the same as the strengths of cubes tested at corresponding ages between plane bearing blocks. The strength was slightly greater for 1 and 3 days ages (maximum 2 per cent) and slightly less at 28 days (maximum 1 per cent). These differences were about the same as the differences between the different batches for a given condition and age. For the ages of 1 and 3 days, when the strengths of the cubes tested between plane blocks were 1560 and 3440 lb. per sq. in., respectively, the difference was slightly greater for the blocks having a mid-ordinate of 0.002 in. than for the blocks having a mid-ordinate of 0.001 in. At 7 and 28 days the strengths were about the same as the strengths of cubes tested between plane blocks, the latter strengths being 4560 and 5610 lb. per sq. in., respectively.

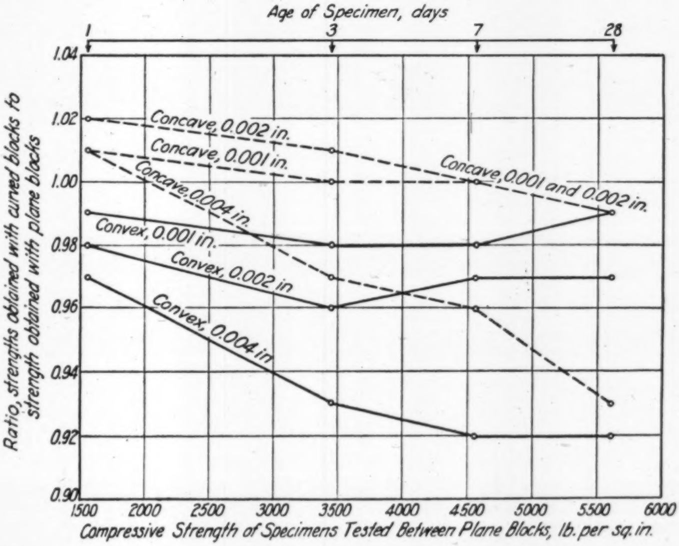

Fig. 1.—Relation Between Curvature of Bearing Blocks and Compressive Strengths of 2-in. Mortar Cubes at 4 Ages.

For cubes tested between convex bearing blocks, having mid-ordinates not exceeding 0.002 in. (loading conditions 5 and 6) the strength was in every case less than the strength of cubes tested between plane bearing blocks (loading condition 1). For all ages the strengths were less for the convex blocks having a mid-ordinate of 0.002 in. than for the convex blocks having a mid-ordinate of 0.001 in. In general, the strength decrease became greater as the age and strength of the cubes increased.

For cubes tested between concave blocks, having a mid-ordinate of

0.004 in. (loading condition 4) the strength at 1 day was slightly greater (1 per cent) than the strength of cubes tested between plane blocks, but the strength decreased progressively with increase in age of cubes and at 28 days was 7 per cent less than the strength of the cubes tested between plane blocks.

For cubes tested between convex blocks having a mid-ordinate of 0.004 in. (loading condition 7), the strength was appreciably less than the strength of cubes tested between plane blocks, and this strength decrease was greater as the age of cube increased, ranging from 3 per cent at 1 day to 8 per cent at 28 days.

The results of these tests are in accord with those of Gonnerman in that, when the curvatures of the bearing surfaces were large enough to cause significant decreases in the strength values, the decreases in these values were greater when the ends of the specimens were convex than when they were concave, and the decreases were greater the greater the strength of the mortar or concrete.

CONCLUSIONS

It is shown that departures from planeness of faces of bearing blocks, in excess of the tolerances now allowed by Tentative Method C 109 - 34 T, but within the limits of the surfaces of some blocks and molds which have been observed, may be responsible for relatively important variations in the compressive strength determinations on 2-in. mortar cubes.

The tests indicate that differences in strength of 3 per cent or more may result from the non-planeness of molds which comply with the requirements of the present Tentative Method C 109 - 34 T, which permit for molds in use a maximum departure of 0.002 in. from planeness of face, and that when the tolerance is reduced to 0.001 in., as now specified for new molds, strength differences up to 2 per cent may be expected as the result of such permissible variation from planeness.

Acknowledgment:

The author expresses his grateful appreciation to H. E. Newcomer and L. A. Messimer, both of the Cement Reference Laboratory staff, who made the tests and calculations, and also to the members of the Bureau staff for their suggestions and their aid in preparing the equipment.

THE INFLUENCE OF LOW CURING TEMPERATURES ON THE HARDENING OF CEMENT MORTARS

BY S. A. MIRONOFF[1]

It is well known from building practice that with the same materials and methods of preparation the hardening process and the final strength of cement mortars depend on the temperature-moisture conditions during the hardening period. At normal temperature and moisture content of the surrounding air, cement-sand mortars increase in strength in inverse relation to the water-cement ratio. With increase or decrease in the temperature of the mass of mortar is involved an acceleration or a retardation in the chemical reaction of cement hydration and in the whole process of hardening. It is known from studies in chemistry that the dissolution of different substances takes place either in direct relation to the temperature, or frequently in inverse relation to it. Consequently, the dissolution of some substances is intensified by temperature rise and that of others, on the contrary, becomes more intense as the temperature decreases. Thus, for instance, $Ca\,(OH)_2$ is dissolved in water at different temperatures, in the following amounts:

At 10 C	0.118 per cent CaO
At 48 C	0.096 per cent CaO
At 70 C	0.076 per cent CaO
At 99 C	0.058 per cent CaO

Colloidal cement (cement glue), which envelops the grains of sand in mortar, forms a gradually hardening gel at a rate which depends on the amount of solvent (water) and on the temperature. It is well known empirically that with the increase of water-cement ratio $\left(\frac{w}{c}\right)$ the rate of strength development and the final strength of the mortars decrease while with temperature rise the rate of strength development increases over a known range. But we cannot be satisfied by a mere empiricism in building problems. This would retard the improvement of the quality of mortars and therefore it is necessary to investigate closely the scientific theory of the hardening of mortars.

Moreover, due to a considerable number of causes which influence the hardening process (water-cement ratio $\frac{w}{c}$, temperature, quantity and brand of cement, aggregates, etc.), generally the part played by any single factor in the development of strength is not determined carefully enough.

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The existing theory of hardening of hydraulic matter, developed by V. Michaelis[2] and based on the hypothesis of swelling, is now considered to be a well-established truth. Doctor Kühl in his report of 1932, in setting forth the Michaelis theory on the hardening of hydraulic mortars, stated that "the inner, or more exactly, the deep layers of cement grains are sucking off water from hydrogel, which process is accompanied by a hydration of these layers. Gel which is on the surface of a grain, envelops it more and more closely until it reaches complete watertightness. Finally, a solid gel is formed from hydrogel." Michaelis attached major importance in the process of the hydraulic hardening of cement mortars to a contracting gel, but besides this he also attached, in his theory, a certain importance to crystalline formations (Fig. 1).

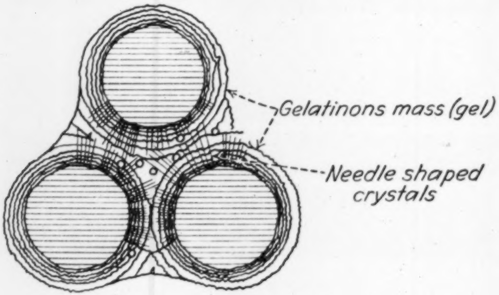

FIG. 1.—Scheme of Cement Hydration and Gel Hardening Processes According to Results of Micro-Analyses.

Many investigators find that the colloidal state of cement mortars presents an intermediate stage. Afterwards it results in the formation of crystalline aggregates which leads to a hardening of mortars with time. This fact is now considered as indisputable.

But notwithstanding a great number of investigations carried out in this direction up to now, there is no complete agreement as to the physico-chemical phenomena taking place during the hardening of mortar. And from existing opinions, constructors cannot wholly explain many practical problems.

One shortcoming of the prevailing theory of cement hardening briefly discussed above is, in our judgment, that the causes affecting the process of swelling of cement mortar gel are imperfectly studied. *It is especially to be noted that serious importance has not been attached to the effect of temperature on the swelling process of cement grains, the formation of gel during this process, and the beginning of its hardening.*

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[2] H. Kühl, "Report on the Theory of Hardening of Cinders," Germany (1931).

Great and perfectly justified attention has been paid to the amount of water which enables one to obtain a definite consistency of mortar and influences very much the chemico-colloidal processes taking place, as well as the hardening. But at the same time the dissolution and swelling of a gel of colloidal particles depend very much on the temperature of the water.

The latter circumstance has not only a theoretical, but also a practical value of some consequence, since mortars and concretes being placed at different seasons are exposed to the effect of different temperatures. The hypothesis we suggest here might, if finally confirmed, modify considerably the opinions in the technology of mortars and concretes and will introduce some absolutely necessary modifications in the theory of cement mortar hardening.

It is our opinion that, in accordance with the theory of swelling, cement particles are hydrating in water, the hydration gradually extending from the surfaces deeper into the grains; that swelling takes place more rapidly at low temperatures (around 2 C.); and that the formation of calcium silicate gel proceeds more slowly at low temperatures, retarding the beginning of crystal formation and of intensive hardening.

Calcium silicate gel of cement mortars takes up a large amount of water and prolongs the time of drawing off water into the grains of cement, so long as the gel is still pervious to water.

In the case where dissolution and swelling with a formation of gel takes place at higher temperatures, this period is considerably reduced due to an acceleration of contraction of the gel on the surface layers of the cement grains. At the same time, besides absorption of water into the grains, intensive evaporation takes place. In many cases the amount of water present in concrete is not sufficient for a complete hydration of the cement.

At higher temperatures, though accelerating the processes of hydration and hardening, no optimum swelling of cement particles takes place, and this influences the final strength. The above considerations suggest the possibility of a scientific interpretation of our expermental results which contradict the opinions prevailing up to now. In checking the method suggested by Prof. I. A. Kireenko[3] for chilling cement-sand mortars after the beginning of the setting of cement for the purpose of developing its higher strength, we have successively lengthened the time of chilling and have obtained an increase of strength up to 50 per cent after chilling at the age of 0.2 to 6 hr. after the preparation.

These experiments are described in detail in the magazine *Stroitelnaia Promishlennost*, No. 12 (1934); see also *Pit and Quarry*, November, 1935, Vol. 28, No. 5, p. 39.

In spite of considerable decrease in concrete strength due to formation of ice crystals on the surface of gravel in the experiments carried out in the cooling room of the Building Research Institute in 1934, when freezing concrete directly after preparation, we obtained higher strengths than those obtained when freezing at the ages of 1 to 3 days and subsequent curing under normal conditions.

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[3] I. A. Kireenko, "Report on Plastering Works During Frosty Weather," Stroitelnaia Promishlennost, No. 8 (1934), U.S.S.R.

The strength of concrete frozen immediately after preparation, later approaches that of concrete cured continuously under normal conditions. Great attention was also attracted by a considerable increase in strength (85 to 35 per cent) obtained in plastic 1:2 portland and puzzolan cement mortars after curing them during the first 7 days at a temperature of 2 C. and afterwards under normal conditions, as compared with the strength of mortars cured continuously under normal conditions. This confirms the above theoretical conclusions and also leads to the conviction that even *after freezing there is no decomposition of cement grains, as stated by Professor Kireenko, but that during the period of freezing and thawing a more considerable swelling of gel takes place on the surface of cement grains in mortar, as well as a supersaturation of gel together with retarded hardening at the beginning.*

Immediate freezing of mortar without subsequent storage for 2 to 6 hr. at lower temperatures gives a somewhat less effect, since the first stage of hydration, which is somewhat lengthened during the freezing period, would not yet be completed. In the process of freezing, an additional destruction of grains is also possible with the increase in volume of freezing water, which penetrates through the capillary cavities of cement particles. This destruction contributes to an increase of the surface of direct ulterior contact between cement and water. The latter circumstance has an opposite effect in the freezing of water in the capillaries of gravel in concrete. Incidentally, it is to be noted that the considerable increase in the strength of mortar, obtained by us as well as by Professor Kireenko, as compared with that of normal specimens in the case where a maximum water-cement ratio was adopted, is related to observed moisture loss from the mortar during freezing. In freezing concretes and mortars at the age of 24 hr. and more, their strength decreases considerably, since the gel has begun to harden. In this case low temperatures contribute in only small degree to swelling, and the frost does not decompose the cement grains but has a destructive effect upon the structure of the hardening mass.

It is therefore quite clear why in most cases after heating of concrete and mortar soon after preparation, we generally obtain a lower ultimate strength than that of concrete and mortar which harden under normal temperature conditions. In the same manner, it is now becoming quite clear why, after 60 to 70 per cent of the designed 28-day strength of concrete has been reached at the age of 3 days by means of electro-heating, we observed afterwards only a small increase in strength and frequently no increase at all for a few days. From this standpoint the practice of electroheating of concrete directly after placing, is wrong; this would result in a lowering of its possible strength. *In any case it is desirable to allow concrete to harden for 24 hr. at temperatures of 15 to 20 C., or for not less than 1½ days at lower temperatures—the latter being even more desirable.*

The existing methods of accelerating the hardening of concrete, with the exception of chemical admixtures and the lowering of water-cement ratio, are based on the use of high temperatures at the very beginning of hardening, whereas our experience indicates that acceleration should involve an exposure at a suitable temperature and for a suitable time before the application of heat. In order to solve this problem as well with respect to the optimum amount of water required, it is necessary to study the problem of moisture evaporation and moisture required for hydration and for a normal formation of gel. The sprinkling of concrete or mortar gives no desirable results.

RESULTS OF INVESTIGATIONS

To check the above statements it is necessary to carry out special experiments for studying the physico-chemical phenomena which take place at the beginning of the mortar hardening, including, first, the formation and hardening of the gel, and second, the consolidation of gel and the strength development of mortar at different temperatures, particularly in combinations of lower temperatures, with further transition to higher ones.

Experiments in curing mortars at various temperatures, studying the phenomena of moisture evaporation and determining quantities of water combined with cement have been carried out which have fully confirmed the above stated principles. Some practical results of our investigations, with a brief description of conditions under which they have been carried out, are here presented.

1. Experimental data published in 1934 by Professor Kireenko concerning the increase in strength of portland-cement mortars after these were subjected to freezing for 3 days at the beginning of the setting period, and afterwards stored under normal conditions, were received rather skeptically among constructors. At the suggestion of K. P. Haidoukoff (Chief Engineer of Chief Building Board at Peoples' Commissariat for Heavy Industry), special tests have been carried out in order to check the data of Professor Kireenko.

Our tests have fully confirmed that having been subjected to freezing, a mortar develops afterwards a higher strength than that of specimens stored under normal conditions.

These tests were carried out using a plastic mortar with portland cement and river sand, at water-cement ratios of 0.7, 0.83, 1.00 and 1.20. The proportions of mixture by volume varied between 1:2.74 and 1:4.55.

Specimens 2.5 hr. old were subjected to freezing for 3 days at temperatures between −15 and −20 C. The setting of paste of normal plasticity began 2 hr. after mixing and was completed in 5 hr., 45 min. After freezing, the mortar specimens (7 by 7 by 7 cm.) were stored together with their corresponding check specimens under moist-temperature conditions, that is, at 17 C. and at a relative humidity of 95 per cent, and were tested after 7 and 28 days of normal storage. In all cases we obtained an increase in strength as compared with corresponding control specimens, the maximum increase taking place in the case of a mortar with a high water-cement ratio $\left(\frac{w}{c}\right)$. The results of compression tests are presented in Fig. 2.

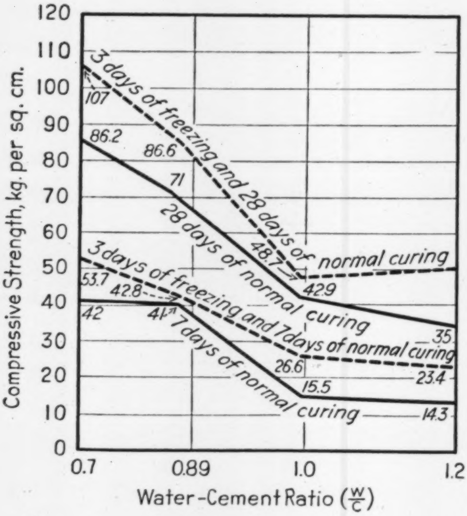

FIG. 2.—Effect of Freezing on the Ultimate Compressive Strength of Cement-Sand Mortars with Different Water-Cement Ratio and Different Curing Conditions.

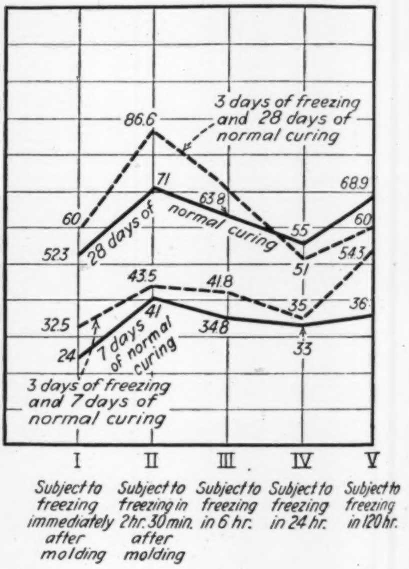

FIG. 3.—Development of Strength in Cement-Sand Specimens in Relation with the Age Before Freezing.

Later we carried out another test using a mortar with $\frac{w}{c} = 0.83$. The specimens were subjected to freezing immediately after molding, after 6 hr., and after 1 and 5 days. In this case, after 7 days of normal storage, we obtained an increase in strength as compared with corresponding check specimens; and at the age of 28 days, specimens subjected to freezing 1 and 5 days after molding showed a decrease in strength. These results are summarized in Fig. 3 which shows that the point of importance is not the moment when the setting of cement begins, but the entire period during which the mortar is in a colloidal state. As mentioned above, we have drawn the conclusion that a positive part is played, not by the freezing process itself and not by the decomposition of cement grains by the freezing water, but by a considerable prolongation of the time during which the mortar remains in a colloidal state at lower temperatures. The latter condition contributes to a greater swelling of cement grains and a greater formation of calcium silicate gel. The subsequent process of hardening (crystallization) takes place more intensively and therefore the mortar shows higher strength.

2. A series of compression tests was made on 7-cm. mortar cubes of 1:2 proportions, water-cement ratio 0.45, plastic consistency, using both portland and puzzolan-portland cements, in which the specimens were stored during the first 7 days at various temperatures, and were thereafter cured for various periods from 1 to 28 days at a temperature of 10 to 12 C. The results are given in the following table:

	Temperature of Preliminary Storage, deg. Cent.	Time of Curing at 10 to 12 C., days	Average Compressive Strength, kg. per sq. cm.	
			"00" Portland Cement	Puzzolan Cement
No. 1	−2	7	15.5	9.4
No. 2	0	7	32.5	31.4
No. 3	2	1	8.0	5.0
No. 4	2	4	25.8	16.6
No. 5	2	7	78.5	45.4
No. 6	2	28	240.0	187.0
No. 7	5	7	46.0	54.7
No. 8	15	1	6.8	7.2
No. 9	15	4	47.5	42.2
No. 10	15	7	63.3	89.5
No. 11	15	28	133.3	136.0
No. 12	30	1	32.0	20.9
No. 13	30	4	89.5	80.8
No. 14	30	7	115.5	133.3
No. 15	30	28	156.4	167.0
No. 16	40	1	31.9	45.8
No. 17	40	4	119.7	144.6
No. 18	40	7	156.9	198.8
No. 19	40	28	216.0	285.3

As we see from the above table, the strength of mortar stored at 2 C. shows at first a slow gain in strength, but from 7 to 28 days it shows almost a double increase as compared with that of mortar stored at 15 C. Likewise, the puzzolan-portland cement mortar stored at 2 C. shows a 28-day strength of 187 kg. per sq. cm. as compared to 136 kg. per sq. cm. for the mortar stored at 15 C., but both of these are considerably lower than the 28-day strength of mortar stored at 40 C. This is to be explained by the fact that puzzolan-portland cements harden slowly at lower temperatures and, inversely, develop their strength rapidly at high temperatures, as well as in the course of time. This example not only agrees well with theory and our other tests, but also permits us to affirm that masonry can be successfully laid in springtime, having in view that warmer days will follow. In fact, all kinds of masonry are constructed in the U.S.S.R. during the whole year without any interruption. In the winter the work is successfully done by means of freezing the mortar which causes no serious trouble.

3. For the purpose of checking whether the hypothesis of the effect of temperature on the swelling of cement grains and the formation of gel during the mixing with water is true, we have carried on a special test, the results of which are briefly described here.

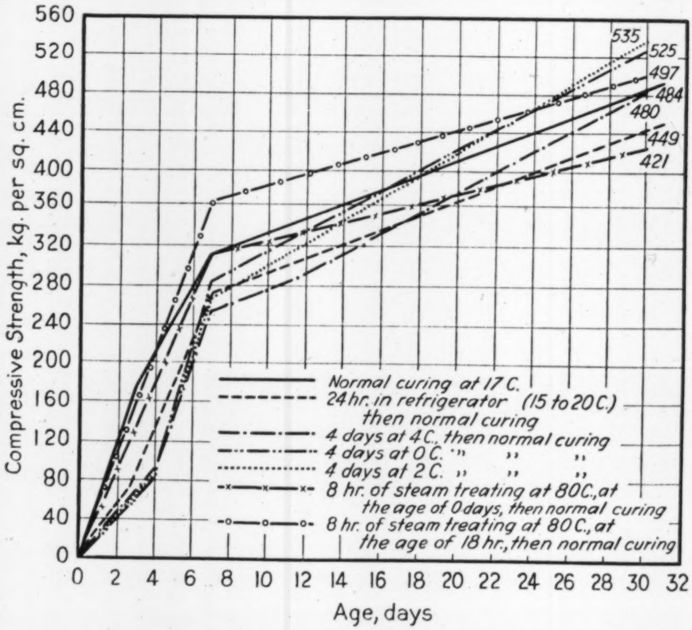

FIG. 4.—Results of Compressive Strength Tests for 1:2 Mortar After Curing at Different Temperatures.

In this test we used plastic 1:2 mortar by weight with a water-cement ratio of 0.40, portland cement of high activity, and the usual river sand. The test specimens were 7-cm. cubes and standard briquets. After placing the mortar in the molds the specimens were cured at 0, 2, 4, 17, 80 (steam treating) and −20 C. The steam treating was begun immediately and also 18 hr. after the molding of specimens, and continued for 8 hr. The freezing continued for 24 hr. in the refrigerator at −20 C. and was begun immediately after the molding. At the temperatures 0, 2 and 4 C. the mortar specimens were cured for 3 days from the time of molding, and were then transferred to the room of normal storage, where they remained until time of testing.

The results of tests are presented in Fig. 4. As would be expected, the strength obtained at the age of 7 days was higher, the higher the temperature of curing during the first days. Afterwards the picture has considerably changed. At the age of 30 and 31 days we have obtained strengths in accord with our theory.

According to the trend of the curves, this relation becomes still more accentuated at later ages. If we consider compressive strengths developed by the cubes at 30 days, we see that assuming the strengths of normal specimens of 487 kg. per sq. cm., specimens subjected to an immediate steam treating have a strength of 421 kg. per sq. cm., and those subjected to steam treating of the same duration in 18 hr. show 497 kg. per sq. cm. Thus, it is seen that it is not expedient to subject a mortar to the effect of high temperatures immediately after placing, as is usually done. The lower strength, developed at the age of 7 days after the initial curing of specimens at 0 and 2 C., has at the age of 30 days exceeded the strength of normal specimens by 40 to 50 kg. per sq. cm.

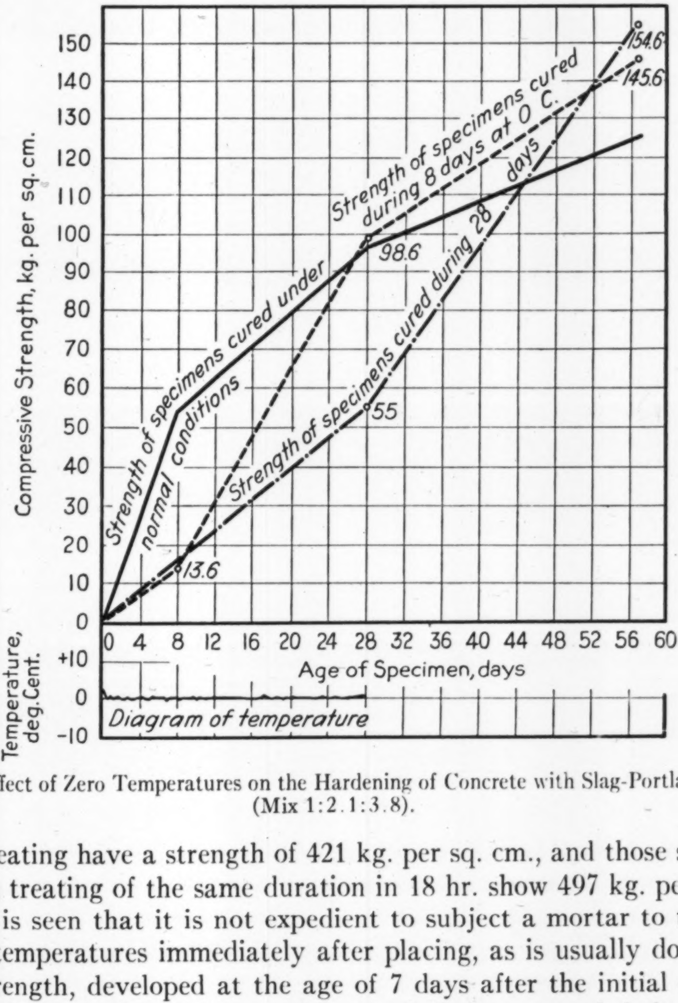

FIG. 5.—Effect of Zero Temperatures on the Hardening of Concrete with Slag-Portland Cement (Mix 1:2.1:3.8).

4. We have now completed tests and prepared a report on the investigations of the effect of different temperatures both above and below zero

on the nature of hardening of concrete with slag-portland cement. As in other tests, standard 20-cm. cubes of concrete immediately after molding were cured for 8, 12 and 28 days at temperatures of 0, 5 and 10 C., and then stored until time of test under normal temperature conditions.

The proportions were 1:2.1:3.8, and water-cement ratio 0.66.

Compression tests of concrete specimens have shown that at low temperatures the early strength is lower than that of specimens hardened under normal conditions (17 C.); and during further curing under normal conditions the strength exceeds that of corresponding check specimens. At the age of 56 days this increase amounts to 20 to 25 per cent. This relation is shown in Fig. 5 which gives the increase in strength of concrete cured for 8 and 28 days at a temperature of approximately 0 C., and then up to 56 days under normal conditions. As is seen from the diagram, the strength developed during an 8-day storage of concrete at 0 C. is 13.6 kg. per sq. cm. as compared with 54.6 kg. per sq. cm. for the corresponding check specimens, that is, the hardening takes place only one fourth as fast as under normal conditions. During 28 days at 0 C. the strength of concrete reached 55 kg. per sq. cm. as compared with 88.7 kg. per sq. cm. for the corresponding check specimens. During this same period, specimens cured under normal conditions, after storage at 0 C. for the first 8 days, developed a strength of 98.6 kg. per sq. cm. as compared with 103.2 kg. per sq. cm. for the corresponding check specimens. At the age of 56 days we obtained most interesting results. The subsequent hardening under normal conditions insured a more rapid increase in strength than the whole period of hardening under normal conditions. Concrete cured during 28 days at 0 C. developed at the age of 56 days a strength of 154.5 kg. per sq. cm. as compared with 119.7 kg. per sq. cm. for the corresponding check specimens. At the same time concrete cured during the first 8 days at 0 C. showed a strength of 145 kg. per sq. cm. as compared with 130.8 kg. per sq. cm. for the corresponding check specimens.

The results obtained are to be explained by the fact that the swelling of slag-portland cement grains during the period of the colloidal state of mortar takes place more intensively at lower temperatures, and therefore the further hardening, the contraction of the forming gel of cement mortar at the period of crystallization, takes its course more effectively.

It is to be noted that tests with slag-portland cement have shown that steam treating at temperatures varying from 40 to 100 C. produces a most favorable effect. The steam treatment in this case has more effect than in the case of concretes with portland cement and even with puzzolan-portland cement. In all cases the strength developed after steam treating exceeds that of corresponding check specimens up to the age of 90 to 120 days. This calls attention to the necessity of a special study of slag-portland cement properties.

5. It is appropriate to refer to the results of tests which have been recently published by O. A. Gershberg[4] concerning the steam treating of 1:3 portland-cement mortars. This author investigated a number of factors including the effect of time of curing specimens before steam treating. The strength at the time of testing was higher the later the specimens were subjected to steam treating at temperatures from 55 to 85 C. As is seen from Fig. 6 the strength of mortar specimens steamed at the age of 2 hr. is only 70 per cent of the strength of specimens steamed at 24 hr. after mixing. At the age of 60 days the strength of mortar subjected to steam treating was found in all cases to be lower than that of specimens cured continuously under normal conditions.

6. It is of interest to note that in steam treatments of specimens at 100 C. for 4 hr., different results were obtained depending on the time at which treatment began after mixing. If the heating is not started immediately, but after 24 hr., the resulting strength is in many cases nearly 50 per cent greater.

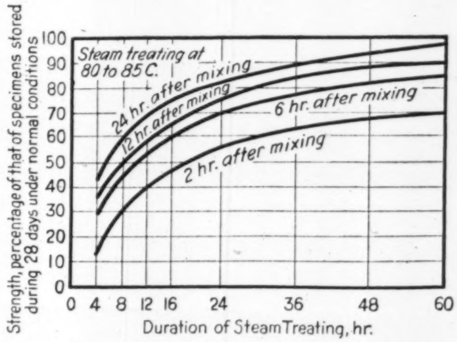

FIG. 6.—Effect of Age (Curing Time) Before Steam Treating on the Resulting Strength of Concrete.

In both cases variations of resulting strength are considerable, but better uniformity and agreement with the results of normal tests is found at the age of 24 hr. after heating. Besides, heating at the age of 24 hr. is done with a gradual rise of temperature, and the specimens are not subjected at once to the action of a temperature approaching the boiling point of water, as is usually the case.

All this prompts us to suggest our method of heating concrete under plant conditions in winter. According to the method proposed by the author, structural elements should not be heated immediately after the placing of concrete, as it is usually done according to specifications, but 24 hr. after placing, especially by low temperature, when the process of cement setting is completed. The use of materials, except water, having a temperature higher than 10 or 15 C. is not recommended, and heating them to high temperatures is harmful. The temperature of water must not be more than 40 C.

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[4] O. A. Gershberg, "Concrete and Ferro Concrete VI," 1935 Edition. Eng. Research Soc. of U.S.S.R.

An important factor is the brand and type of cement. This together with the lowest temperature occurring during curing generally determines the time at which heating should be started. In each particular case the beginning of heating is to be determined by strength requirements, by the reuse of forms and the permissible duration of curing in heating rooms (at a plant), by considerations of economy, etc. Under plant conditions, opportunity is afforded to adopt a combination of curing temperatures, that is, to cure concrete and reinforced concrete elements at first at lower temperatures, and then to subject them to heating. This method is very important in manufacturing small products where sand concrete is to be used.

The heating is to be done with a gradual increase in temperature. In winter, lest concrete should be frozen before the heating, it is necessary to subject it at first to a low level heating. When portland cements are used it is not necessary to subject them to heating at a level higher than 50 to 60 C., while in using puzzolan and slag-portland cements it is preferable to raise this level up to 70 to 80 C., which at once reduces the time of heating. Our tests have shown that in the case of most cements, in order to obtain 70 per cent of 28-day strength it is sufficient to continue the heating during 24 hr. and then to allow the concrete to cool slowly. Such a percentage is obtained less easily when portland cements are used. It is not desirable to heat reinforced-concrete elements immediately at high temperatures in consequence of an observed decrease in bond strength between steel and concrete.

The method proposed by the author according to which the heating of concrete and reinforced-concrete elements is to be begun at later ages, will on the basis of the same materials insure a higher strength of concrete, which of course is our purpose.

Analogously, accelerated laboratory methods of testing cements and concretes should be revised and modified in the direction of delaying the time of heat application. In this way, only the period of hardening will be accelerated, but not the period of setting. Thus will the natural strength development in the course of time be approximated.

DISCUSSION

MR. P. H. BATES.[1]—I had an opportunity to review this paper prior to its presentation, and being interested by the theories of the author, I started some few tests which can be presented here. The results are given in the accompanying Fig. 1.

From this it will be noted that 3 by 6-in. concrete cylinders were made and stored under various conditions for different ages in hermetically sealed metal molds. Certain of these were kept at 37 F. (slightly above freezing) for one, three, five, or seven days before being submitted to either a treatment at 150 F. for six days or in an autoclave at 120 lb. pressure for a day. A companion series was given the same later treatments after initial curing at 70 F. A few cylinders were also tested after curing for their entire age at 70 F. The heat of hydration at the longest testing age was also made as indicated in the table. Hence the conditions of curing indicated by the author were used as well as certain additional ones.

Just as it has been noted before and can again be noted here, in steam curing of concrete the effect will depend upon some unknown property of the cement and hence different cements give different results, so in this low temperature initial curing different results were obtained for the different cements. In the case of the standard portland cement no differences resulted when the concrete was subjected to the low or high temperature initial curing when the later curing was at 150 F. Under these conditions also the strengths were approximately the same as though the concrete had been stored at all times at 70 F. But the autoclave treatment more than doubled the strength, without again showing any difference in the low or 70 F. initial curing. While with the two other cements the effect of the two different initial curings was similar after later curing at 150 F., the autoclave treatment showed that initial curing at the low temperature gave from 500 to 1500 lb. per sq. in. more strength than initial curing at 70 F. Hence it can be seen that the author's hypothesis and results are corroborated strikingly with one of the three cements, to a lesser degree with a second, and not at all with a third.

The heats of hydration, which were all made after 13 days of hydration, do not show anything very significant, except that the actual heats in calories are no indication of the strength. It is to be noted, however, that all of the special treatments show by the heats of hydration a greater degree of hydration than when the cement was maintained at 70 F. at all times.

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[1] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

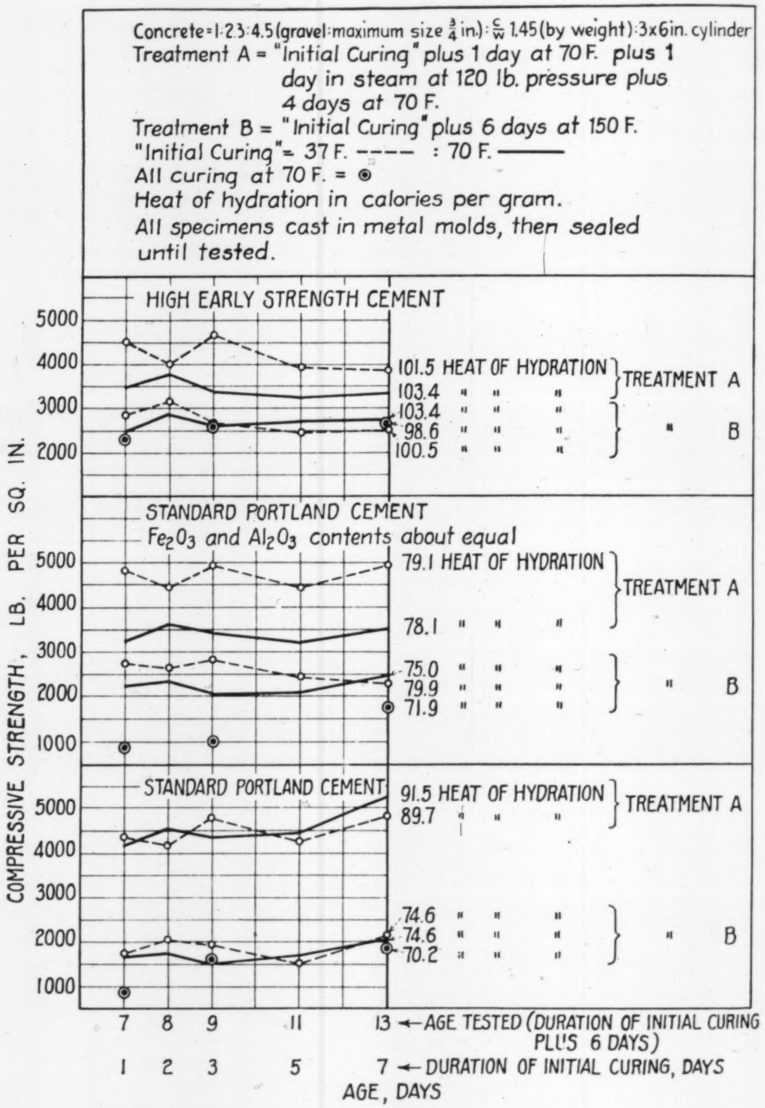

FIG. 1—Effect of Low Temperature Curing on Concrete.

MR. D. S. COLBURN.[2]—I am interested in what Mr. Mironoff found where it can be applied in a practical way. There came to my mind, for example, an experience I had in attempting to finish a concrete pavement at about 35 F. It could not be done. In Russia they may be able to control the temperature so as to keep it down around freezing while the concrete is curing. In this country we cannot do that and I would hesitate to suggest to our sales department any thought that concrete placed at freezing is going to be better than concrete placed at 70 F. It costs a lot of money to attempt to bring temperatures down, but it does not cost much to bring them up.

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[2] Vice-President, Marquette Cement Manufacturing Co., Chicago, Ill.

RATING THE CHARACTERISTICS OF FRESH CONCRETE

By Harmer E. Davis[1] and J. W. Kelly[1]

Synopsis

As an aid in the judgment of the quality of fresh concrete, and as a supplement to physical tests, this paper discusses a systematic method for visual observation of the various factors—consistency, plastic characteristics, apparent composition of mix, tendency toward segregation, and workability in general. The use of a numerical rating scale in evaluating the quality of different mixes with regard to handling and placing is also discussed.

Introduction

It is desirable that fresh concrete be relatively easy to mix, transport, deposit, compact, and finish and that it remain homogeneous during the period of placement. The composite quality sought depends upon a number of factors some of which cannot be satisfactorily measured. In practice, the general suitability of concrete for a given purpose is evaluated largely through visual inspection. The object of this paper is to discuss the visual inspection of characteristics of fresh concrete and the rating of factors related to workability and general suitability.

It is believed that information regarding the characteristics of fresh concrete, based upon a systematic method of visual inspection, should occupy a more definite place in the record of concrete tests than is now customary. Such information, as a supplement to the results of commonly made tests, provides a uniform basis for comparing the qualities of various concretes, particularly in extensive series of trial-mix tests, and serves as a guide to correct faulty mixes or to explain irregularities in the results of subsequent tests.

Visual Inspection of Characteristics

An estimate of the quality of fresh concrete by visual inspection is necessarily based upon characteristics that are readily observable. Although some observable characteristics are partly or entirely the effect of others, causing some apparent duplication, each angle of approach is of value as evidence regarding quality. The results of any physical tests made at the time of observation also constitute valuable evidence;[2] however, in general the discussion of this paper is limited to visible characteristics.

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[1] Assistant Professor of Civil Engineering, and Research Engineer, Engineering Materials Laboratory, respectively, University of California, Berkeley, Calif.

TABLE I.—CHECK LIST FOR RATING OF CHARACTERISTICS OF FRESH CONCRETE.

NOTE.—On the following form, check or encircle the appropriate word applying to each property. If conditions warrant, the word at either end of the scale may be qualified further by adding such words as "very", "moderately", or "slightly" in the blank space immediately preceding the word.

Project..
Mix.............................. Batch No............ Observer..................
Date........................ Hour................ Time After Mixing................
Temperature........ Relative Humidity........ Mixing Conditions....................
Depth and Area of Layer................ Method of Compaction........................

	RATING NUMBER
1A. CONSISTENCY: dry stiff medium wet sloppy a. Slump.................... in.; b. Flow.................... per cent	
1B. STIFFENING: Remarks.. a. Rate:................ rapid normal .... slow b. Amount:.. c. Apparent cause: evaporation absorption chemical action	
2. PLASTIC CHARACTERISTICS: a. Cohesiveness:.................... tacky normal.............. short b. Resistance to indentation:......... hard firm.................. soft c. Plasticizing component:......... smooth balanced............ harsh	
3. APPARENT COMPOSITION OF MIX: a. Cement content:.................... lean O.K. .... rich b. Consistency of paste:............... thin O.K. .... thick c. Character of cement:............. gritty O.K. .... sticky d. Proportion of sand:......... undersanded O.K. ...... oversanded e. Gradation of sand:................ grainy O.K. .... earthy f. Gradation of coarse aggregate ..... rocky O.K. .... pebbly	
4. SEGREGATION: a. From pile on flat surface: none slight considerable excessive b. In form: none slight considerable excessive (Type..) c. Water gain: none slight considerable excessive (If water gain occurs: .... slow .... rapid) d. Laitance: none slight considerable excessive	
5. WORKABILITY: a. Operator's visual rating:................ good fair............ poor b. Time to compact............ (specimen) by vibration:............ sec. (Type and size of vibrator............ ; frequency...... r.p.m.) c. Finishing qualities:..................... good fair............ poor	
6. SUITABILITY RELATIVE TO CONCRETE FOR.................. a. Rating: .... good fair .... poor — QUALITY INDEX:	

7. SUGGESTIONS FOR IMPROVEMENT..

The major factors to be considered are (1) consistency, (2) plastic characteristics, (3) apparent composition of mix, (4) tendency toward segregation, and (5) workability. For purposes of observation, these factors are subdivided as shown in Table I, which is a check list adapted from a form in use at the Engineering Materials Laboratory of the University of California.

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[2] For a recent discussion of the measurement of physical properties of concrete, see "Factors of Workability of Portland Cement Concrete," by W. H. Herschel and E. A. Pisapia, *Journal*, Am. Concrete Inst., May–June 1936; *Proceedings*, Vol. 32, pp. 641–658 (1936).

The check list tends to insure a more adequate record than is obtained when an observer merely comments upon some factor which may be apparent to him at the moment. It also serves to aid inexperienced men to know what to look for in a mix.

The check list is reasonably complete; however, for many purposes it will be unnecessary or impracticable to consider all of the items listed. Comments on the various items follow.

Consistency:

Herein the term consistency refers to the degree of wetness or dryness of the mix—the familiar concept in concrete practice. Descriptive terms are "dry", "stiff", "medium", "wet", and "sloppy". The slump and flow-table tests serve well to classify concretes as to their degree of consistency; however, on the check list it may be desirable to supplement the test data with a notation as to the appearance.

Inasmuch as concrete stiffens with time after the materials are wetted, observations of consistency should be made at known—and preferably controlled—times after mixing. If the stiffening of concrete appears to be abnormally rapid, effort should be made to determine whether it is due to (*a*) evaporation of mixing water, especially if the concrete is exposed to wind or sun; (*b*) absorption of mixing water by aggregates; or (*c*) action between the cement and water.

Plastic Characteristics:

Although a complete definition of plasticity involves the yield value and the mobility,[3] for the purposes of visual inspection of fresh concrete the observable characteristics are (1) cohesiveness; (2) resistance to indentation, or hardness; and (3) smoothness, or the degree to which the plasticizing component is present.

Cohesiveness.—Cohesiveness is that property by virtue of which the particles in a mass tend to hold or stick together; the opposite of cohesiveness is dissociation or separation of particles. Some cohesiveness is desirable in order that a concrete can be handled with minimum tendency toward segregation; on the other hand, too high a degree of cohesiveness produces a tacky mix with resulting loss of workability. One indication of the degree of cohesiveness is obtained by lifting a shovel or trowel flatwise from the smoothed surface of concrete—the greater the cohesiveness the greater the effort required to lift the tool and the more roughened the concrete surface. The opposite of *tackiness* (high cohesiveness) is taken as *shortness*.

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[3] Tentative Definitions of the Terms Consistency and Plasticity (E 24 – 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1245 (1934); also 1935 Book of A.S.T.M. Tentative Standards, p. 1439.

Resistance to Indentation.—*Hardness* or *softness* may be judged by troweling or ramming the surface of partly compacted concrete and estimating the relative resistance to indentation.

Presence of Plasticizing Component.—The apparent sufficiency in concrete of an element—either paste or mortar—which imparts a distinct lubricating or plasticizing quality to the mass is called *smoothness*; and the lack of such an element is called *harshness*.

Apparent Composition of Mix:

The appearance and "feel" of the mix while it is being worked (dumped, spaded, or finished) frequently indicates whether the proportions are satisfactory or whether the composition is defective. Usually it is best to look for abnormalities in the mix; otherwise the mix should be classified as satisfactory. The following factors are observable.

Cement Content.—The mix may appear to be too *lean* or too *rich*. Efforts should be made to distinguish between lack or excess of cement and lack or excess of fines in the sand.

Consistency of Paste.—The paste may be so *thin* that it fails to cover the aggregate particles adequately, fails to impart proper "body" to the mass, or tends to separate from the pile. On the other hand, the paste may be so *thick* that it will not lubricate or "carry" the aggregate. Effort should be made to distinguish between (1) characteristics caused by the consistency of the paste, (2) those caused by the amount of paste per unit of aggregate, and (3) those caused by the presence of fine sand and silt in the aggregate.

Gradation and Character of Cement.—A cement which is too coarsely ground may fail to lubricate the mass properly, resulting in a *gritty* mix. On the other hand, the use of cement ground extremely fine may result in a *sticky* mix. Further, some cements impart a definite "fatness" to the mix; the distinct presence or absence of this quality should be noted on the check list.

Proportion of Sand.—For every mix there is presumably an optimum sand content which results in maximum plasticity and homogeneity. If the mix is *undersanded* it will tend to be harsh and may result in honeycombing. On the other hand, if *oversanding* is extreme, an inefficient relationship between cement content and strength may result; further, the mortar component of the concrete is likely to be undercemented and therefore porous. The optimum content of mortar (sand plus cement-water paste) appears to be that which overfills the original voids in the coarse aggregate by a margin just sufficient to produce a reasonable degree of mobility.

Gradation of Sand.—The use of sand which is too coarse—either having an excess of some coarser size or lacking in fines[4]—results in a *grainy* mix of poor workability and in water gain of concrete. Although in the wetter consistencies a sand which contains a large amount of fines tends to produce a smooth-working mix, in the drier consistencies an excess of fines produces an *earthy* appearance and tends to deaden the mix. The optimum percentage of fines in the sand is probably just above the point at which the mix ceases to be noticeably grainy. Effort should be made to distinguish between an excess of fines in sand and an excess of cement in the mix.

Gradation of Coarse Aggregate.—Usually considerable differences in gradation of coarse aggregate are permissible in an otherwise well-proportioned mix. However, an excessive amount of the largest size group results in a *rocky* mix from which these particles tend to work out; and an excessive amount of the pea-gravel size (about $\frac{3}{8}$ in.) results in a *pebbly* mix which is difficult to compact and finish satisfactorily. Effort should be made to distinguish between the results of faulty gradation of coarse aggregate and apparent rockiness due to undersanding.

Segregation:

In wet or undersanded mixes of low cohesiveness, there is a tendency for the coarse aggregate and mortar to separate during the period of handling and compacting. Evidence of segregation may be obtained by examination of concrete as it is dumped in a pile and after it is placed in the forms; it is particularly desirable to examine vertical surfaces after form removal for evidences of segregation during the period of placement.

Water gain and *laitance* are special forms of segregation. Water gain is evidenced by the formation of small pools of clear water on the surface of concrete which has been left undisturbed for a quarter hour or so after being placed. Observation of the formation of laitance—the scum containing fine non-cementitious particles of cement and silt which rise to the surface of wet mixes when subjected to spading, troweling, or vibration—should not be overlooked, although the appearance of laitance is not always evident immediately after the mix is placed.

Workability:

Herein the use of the term workability is restricted to describe the inverse of the effort required to mold or form concrete, that is, to work it from one shape to another. The Powers remolding test incorporates the essential features of a satisfactory workability test,[5] and certain methods of vibratory compaction in a fixed form of mold offer considerable promise.

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[4] Particles passing the No. 50 sieve.

[5] T. C. Powers, "Studies of Workability of Concrete," *Proceedings*, Am Concrete Inst., Vol. 28, pp. 419-448 (1932).

As no workability test has been generally accepted, any physical measure of workability should be supplemented by a description of the visible characteristics of the concrete. Workability is influenced by the factors previously discussed, and an estimate or rating of workability should be based upon a general consideration of those factors, together with an estimate of the readiness with which the material can be consolidated into place.

RATING OF QUALITY

The rating of the quality of a concrete mix relative to handling and placing involves, in addition to observing the apparent characteristics as already described, a consideration of the suitability of the concrete for the given type of construction. To make a satisfactory rating, the observer should keep constantly in mind the probable behavior of the mix under the given field conditions. A mental standard must be established; naturally, experience and judgment are prerequisites.

Sometimes the rating of each major characteristic of concrete for a particular purpose can be adequately expressed by means of a descriptive term such as "good," "fair," or "poor." On the check list (Table I), space is provided for such rating of the apparent workability and the general suitability of concrete for a given purpose.

In judging the relative quality of concretes there appear to be some advantages in building up a composite rating or index from a consideration of the several individual characteristics. Not only are mixes of high all-round quality distinguished from those of low quality by such an index, but also, in the attempt to secure the composite rating, the observer may detect defects which can be corrected.

Numerical Rating Scale:

For many purposes it is desirable to express the degree of quality in a quantitative manner. A rating scale employed by the authors in tests of concrete, adapted from scales employed by others but extended so as to be of more or less general application, is as follows:

10	Perfect
9	Excellent
7	Good
5	Fair; average
3	Poor
1	Very poor
0	Zero

The scale is easily remembered as the classifications are few and the numbers small; yet the scale has a sufficiently wide range to express differences in quality which are readily apparent to the ordinary observer. Only rarely will it be necessary to use 0 or 10; for most purposes the scale from 1 to 9 covers the range.

On the check list, spaces are provided in the right-hand column for rating numerically the major characteristics; if desired, some or all of the subordinate characteristics may also be rated.

By means of a numerical scale, not only can results of observations of a single characteristic or type of test be compared but also composite indices, based upon several properties, can be computed by averaging the individual rating numbers. Sometimes it is possible to assign weights for properties which are particularly desirable or important. Care and judgment must be exercised in the choice of properties or characteristics that are significant. The final rating number which includes the various separate ratings is herein called the "quality index" for the given set of conditions.

TABLE II.—COMPARATIVE RATINGS OF CONCRETES CONTAINING 0 TO 6-IN. AGGREGATE AND HAVING A CEMENT CONTENT OF 1.0 BBL. PER CU. YD.

	Mix A	Mix B	Mix C
MIX DATA			
Proportion of sand to coarse aggregate	30:70	35:65	25:75
Water-cement ratio, by weight	0.60	0.70	0.49
Slump, in.	$3\frac{3}{4}$	$3\frac{1}{2}$	$\frac{1}{4}$
RATING OF FRESH CONCRETE			
Consistency (for mass concrete)	7	7	3
Fatness	9	7	3
Apparent proportion of sand	8	5	3
Tendency toward segregation of aggregate	9	9	3
Water gain on 18 by 36-in. cylinders (by test)	7	3	9
Time for vibratory compaction in 18 by 36-in. cylinder	9	7	3
QUALITY INDEX	8	6	4
Observer's general comments	O.K; too wet	Slightly oversanded; too wet	Undersanded; dry
RESULTS OF SUBSEQUENT TESTS			
Compressive strength at 28 days, lb. per sq. in.	2430	1720	3570

For some characteristics, as conditions change progressively the rating changes in the same direction throughout the range. However, for characteristics in which an optimum is exhibited the rating number increases up to the optimum and then decreases as conditions continue to change. For example, a mix exhibiting little or no water gain would receive a high rating and with increasing water gain the rating would become progressively lower; on the other hand, with regard to proportion of sand in the mix, either less or more sand than the apparently optimum amount would result in a lower rating.

In any system of rating it is important to have standards of reference which should be clearly defined, and which should remain constant over a period of time. The use of photographs showing examples of a given characteristic in each of several ranges of quality, together with the rating for each range, has been found advantageous by the authors in preventing what might be called "drift" in the observer's standard for rating. Stereophotographs have been used to special advantage for some types of tests.

As has been pointed out, the rating of qualities pertaining to handling and placing can be made only when the intended use of the concrete is considered. For instance, a moderately stiff mix might receive a high rating for consistency if the concrete were intended for use in massive construction, whereas the same mix would receive a low rating for consistency if it were intended for use in narrow reinforced members.

By way of illustration of the determination of the quality indices of concrete mixes, the data shown in Table II have been taken from a series of trial-mix tests for mass concrete. A limited number of characteristics were selected for rating. The computed quality indices served as a basis for selecting a limited number of mixes for further detailed investigation. Although no statistical studies have been made of the closeness of agreement in rating as between different observers, trained observers have seldom differed materially in rating a given property for a particular concrete.

CONCLUDING REMARKS

In conclusion, it is believed (1) that systematic inspection and record of the characteristics of fresh concrete are desirable adjuncts to the tests; and (2) that a method of numerical rating, based upon the requirements of the particular work and perhaps weighted to take account of important characteristics, permits at least a rough quantitative comparison of concretes which may be made of different materials and at different times and places.

THE EFFECT OF TESTING SPEED ON STRENGTH AND ELASTIC PROPERTIES OF CONCRETE

By Paul G. Jones[1] and F. E. Richart[1]

Synopsis

Tests were made to study the effect of speed of testing upon the compressive strength and the stress-strain relation for plain concrete. Tests were made on three grades of concrete, at ages of 7 and 28 days. Loading of a 6 by 12-in. cylinder to failure was accomplished in periods of 1 sec., 5 sec., 20 sec., 1 min., 2 min., 10 min., 30 min., 1 hr., and 4 hr., with a corresponding rate of stress application ranging from 3870 down to 0.12 lb. per sq. in. per sec. Both load and strain were measured by means of telemeter gages of the carbon resistor type. For all but the very slow loading rates, autographic records of load, strain and time were taken by means of an oscillograph recorder, equipped with super-sensitive elements, which provided satisfactory precision of measurement.

The test results indicate that the strength increases with increase in speed of loading, the ratio of strengths for the highest and lowest rates used being about 1⅓ to 1. The secant modulus of elasticity at 90 per cent of the maximum load also increased with increase in rate of loading. This indicates that a part of the measured strain in the tests is due to creep; the amount of this creep increases with the length of time involved in the test. Additional data indicating that the amount of creep is not proportional to the intensity of stress were obtained from a few special tests, in which the oscillograph record permitted observations of creep from the instant at which a load increment was applied.

Object of Studies

The purpose of the tests described here was to investigate the effect of varying the testing speed upon the strength and other properties of concrete. While the loading rate has been recognized as a factor affecting the strength of materials in general for many years, very few studies have been made which applied specifically to concrete. Of the tests made in the United States, those of Abrams **(1)**[2] in 1917 were probably the first. He determined a definite relation between rate of loading and compressive strength of standard concrete cylinders. Tests in 1930 at Lehigh University **(2)** and the University of Illinois **(3)** on reinforced concrete columns included two rates of loading, one rapid, the other consisting of intermittent loading, with increments of load applied 4 hr. apart. In 1930, Glanville **(4)**, in England, made tests and evolved a hypothesis as to the relation between speed of loading and the shape of the stress-strain curve for concrete in compression. He indicated that the amount of plastic strain decreased as the rate of loading increased. Slater (5) made tests in 1931 in an attempt to secure data bearing on Glanville's theory, but the tests were too few to be conclusive. In 1933, Withey (6) directed tests to determine the relation between rate of loading and the flexural strength of concrete.

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[1] Test Assistant in Theoretical and Applied Mechanics, and Research Professor of Engineering Materials respectively, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 391.

The present series of tests had a two-fold object: (*a*) to study the relation between rate of loading and the compressive strength of concrete and (*b*) to observe the effect of rate of loading on the stress-strain relation for the same concrete in compression. The work started as a project of the Society's Committee C-9 on Concrete and Concrete Aggregates and was made possible by the active sponsorship of the Section on Effect of Speed of Testing of Committee E-1 on Methods of Testing. Financial support of the work was provided through a grant of funds by Committee E-9 on Research.

Acknowledgment:

These tests were carried on as regular research work of the Engineering Experiment Station, University of Illinois, in cooperation with the American Society for Testing Materials. The Society was represented by an Advisory Committee consisting of Prof. M. O. Withey, chairman, Mr. P. G. McVetty and Mr. D. E. Parsons. The project was under the administrative direction of Dean M. L. Enger, Director of the Engineering Experiment Station and Prof. F. B. Seely, Head of the Department of Theoretical and Applied Mechanics at the University. Acknowledgment is also due to Mr. O. S. Peters, Washington, D. C., who furnished the telemeter apparatus used in measuring loads and deformations.

Outline of Tests

The program of tests was simple. Standard 6 by 12-in. cylinders, of nominal strengths of 2000, 3500 and 5000 lb. per sq. in. were made and three or more were tested at the ages of 7 and 28 days at nine testing speeds. For the 7-day cylinders of 2000-lb. concrete, the time intervals in which a cylinder was loaded to failure were roughly as follows: 1 sec., 5 sec., 20 sec., 1 min., 2 min., 10 min., 30 min., 1 hr. and 4 hr. The rates of loading for the other grades of concrete were similar, but varied due to differences in stiffness and strength of the concrete. A few additional cylinders were made for special tests on the creep of concrete.

Test Cylinders:

All of the cylinders were made with Wabash River sand and gravel and Marquette Portland Cement. The sand was a well-graded torpedo sand, of fineness modulus 2.95. The gravel was of 1-in. maximum size and fineness modules 6.50. The mixtures used to produce the three grades noted above were:

NOMINAL STRENGTH, LB. PER SQ. IN.	PROPORTIONS, BY WEIGHT	WATER-CEMENT RATIO, BY VOLUME
2000	1:4:5	1.4
3500	1:3.25:4	1.1
5000	1:2.25:2.75	0.8

The cylinders were machine mixed, and made in lots of five. They were cured in a standard fog room at 70 F. and were tested moist.

Description of Tests:

The tests were made in a 300,000-lb. capacity Olsen screw-power machine. This machine was geared to produce speeds of the moving head, running idle, of 0.05, 0.3, 1 and 6 in. per min., which were used for the higher rates of loading. Slower speeds were provided by connecting up to a variable-speed direct-current motor; this served for all but the 4-hr. tests, in which it was necessary to build up the load in small increments, using the slowest motor speed available.

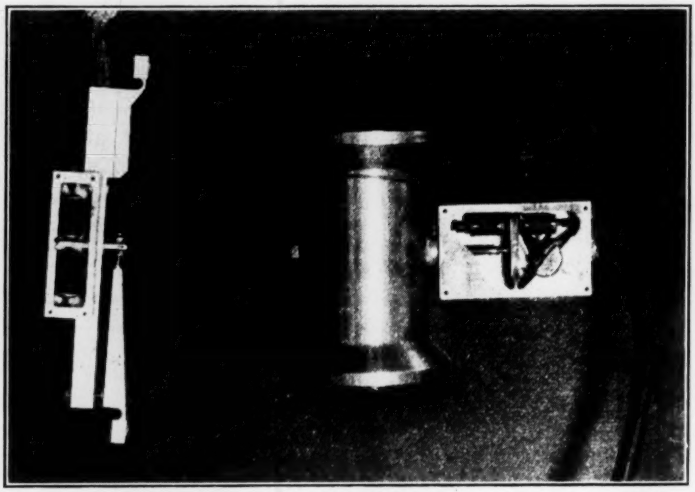

FIG. 1.—View of Telemeter Gage and Loading Block, with Carbon Pile Elements Exposed

Concrete strains were measured by means of two carbon resistor type double-stack telemeter gages[3] of 8-in. gage length. The essential working part of the telemeter consists of a series of contacts between carbon disks or rings, which when displaced produce a corresponding change in electrical resistance. This change of resistance is utilized in a simple form of Wheatstone bridge circuit.

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[3] The theory of operation of the telemeter is described in detail in the following papers:

Burton McCollum and O. S. Peters, "A New Electrical Telemeter," National Bureau of Standards *Technologic Paper No. 247* (1924).

O. S. Peters and R. S. Johnston, "New Developments in Electric Telemeters," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 592 (1923).

The load on the cylinder was also measured by a telemeter-type loading block, similar to the strain gages except that the displacement of the carbon stacks was produced by the lateral deformation of a steel cylinder which transmitted the load (see Fig. 1).

The Wheatstone bridge circuits of the two strain gages and the loading block were contained in a three-element panel board, connected in parallel and adjusted to the desired voltage by means of a series resistance. Direct current from storage batteries, at a constant potential of 6 v., was used in all tests. The extent of unbalance of the Wheatstone bridge circuits caused by movement in the gages was transmitted to an oscillograph recorder, or, by means of a transfer switch, indicated on a milliammeter on the panel board where it could be read directly. Visual readings were taken on the slow-speed tests requiring 30 min. or longer.

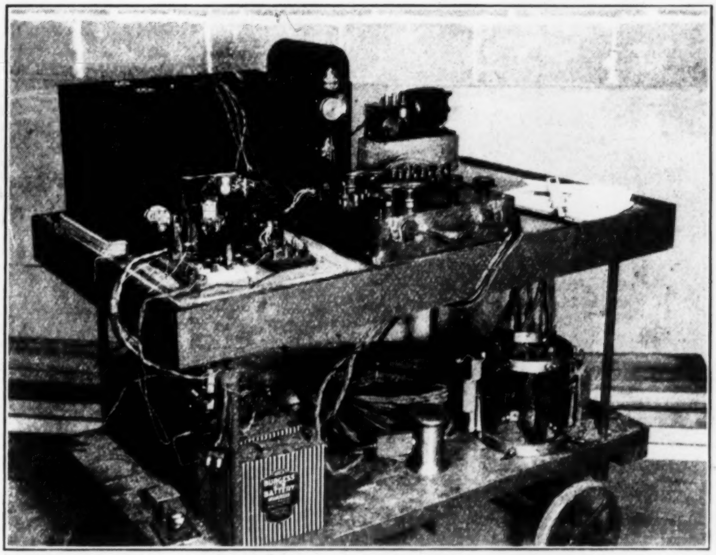

FIG. 2.—View of Oscillograph, Panel Board, Chronograph, Batteries and Gages.

The four-element oscillograph used was equipped with super-sensitive elements of the oil-well type. The camera recording the deflection of these elements was driven by a back-geared, 12-v. d-c. motor to provide recording speeds of 0.01 to 25 in. per sec. The 6 to 8-v., 2.5-amp. gas-filled oscillograph lamp gave sufficient intensity of light to register on grade A Insurance Bromide paper used for the high-speed tests. The timing wave on the oscillograph record was produced by an electromagnet excited by impulses from a chronograph at intervals of 15 sec., 1 sec., or $\frac{1}{5}$ sec. or by 60-cycle alternating current, as required by the speed of the test. A vacuum tube was used to amplify the pulsations produced by the chronograph. Figure 2 shows a view of oscillograph, panel board, batteries, telemeter gages, etc.

The telemeter strain gages were designed for a movement of ±0.01 in., but were adjusted to increase the range in compression to 0.015 in. The gages were clamped to two steel rings attached to the cylinder 8 in. apart, and provided with positive stops to protect the gages when the test cylinder failed (see Fig. 3). The gages were calibrated frequently by use of a micrometer microscope, suitably mounted. At low strains the calibration curve was a straight line; at high strains it deviated somewhat. It is to be noted that the principal use of the strain measurements, the determination of the modulus of elasticity of the concrete, was in connection with the smaller strains where the calibration curve was straight. The sensitivity of the telemeter gage was about 0.00004 in. total deformation per division on the milliammeter or per 0.01 in. ordinate on the oscillograph record, which was the smallest unit observable. With the 8-in. gage length used, this corresponds to a sensitivity of 0.000005 in. per inch, and the error in reading may have been approximately of this magnitude. The sensitivity

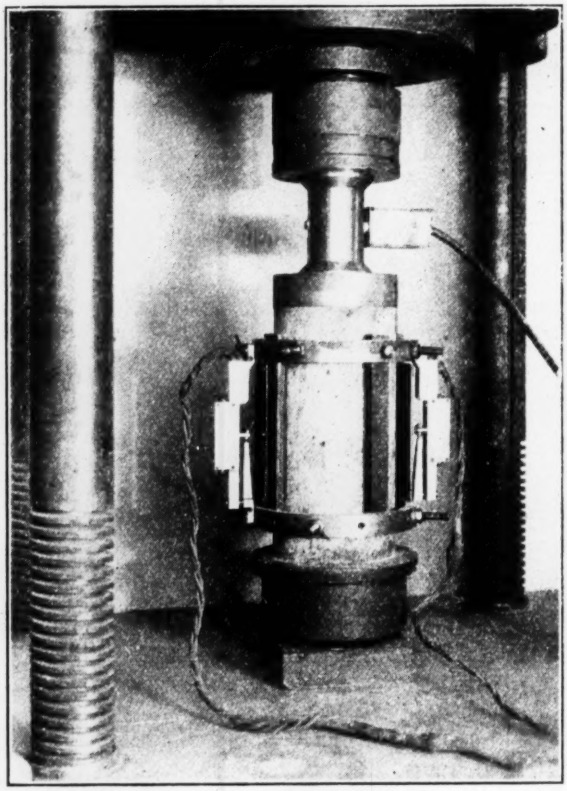

FIG. 3.—Telemeter Gages and Loading Block Mounted on Concrete Test Cylinder.

Note spherical block above and below test piece.

of the load-measuring block was about 500 lb. per division on the milliammeter and 250 lb. per 0.01 in., ordinate on the oscillograph record. The smallest reading in terms of average cylinder stress was thus about 20 lb. per sq. in. by visual reading and 10 lb. per sq. in. by oscillograph. For the higher loads this sensitivity was decreased by means of shunts across the milliammeter and oscillograph elements. The sensitivity of the oscillograph elements as noted above could, of course, be changed considerably by changing the tension of the element strips.

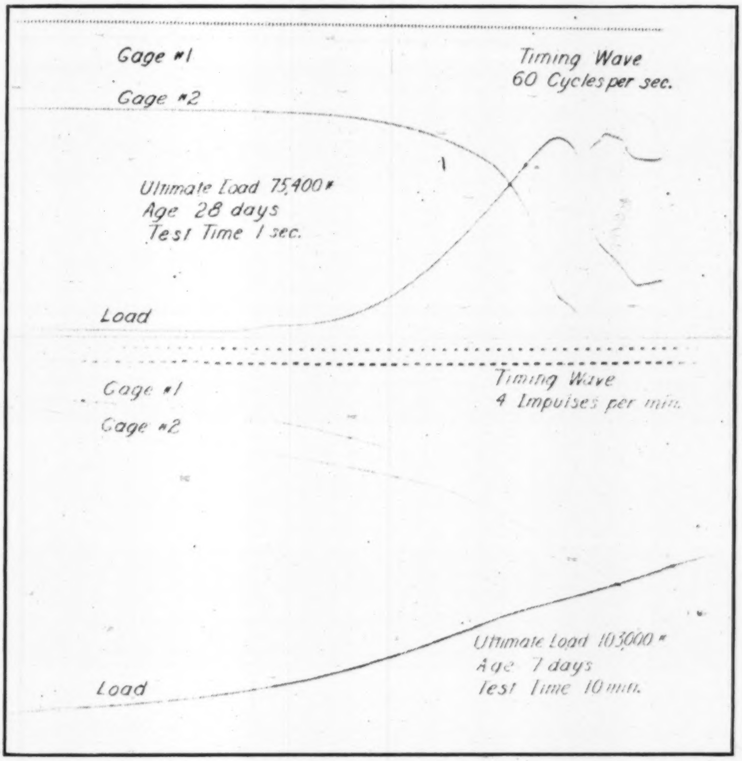

FIG. 4.—Oscillograph Records, Showing Two Strain Curves, Load Curve and Timing Wave.

Tests were made to determine whether an error in load reading might be introduced by a change in temperature of the load-weighing block due to rapid application of load. A copper-constantan thermocouple was clamped to the surface of the block and insulated from the air; this was connected to a portable potientiometer capable of indicating a temperature change of $\frac{1}{2}$ F. or less. With load applied to produce failure of a concrete cylinder at the highest of the testing speeds, there was no observed temperature change in the loading block. Since the telemeter is so attached to the block that it should be very nearly self-compensating for the effect of temperature changes, as shown in Fig. 1, it is believed that any errors in load measurement due to this cause are negligible.

It was also noted that the instrument reading for the load-weighing block did not always return exactly to zero when the load was removed. However, the block was calibrated frequently, and in the same testing machine that was used for the concrete tests. There was always a slight lag at the load of 4000 lb., where the weight of the screws of the testing machine was picked up and this telltale lag appeared on the test records. This point on the curve was used in verifying the load readings, where there was any doubt as to the zero point on the calibration curve. With this independent check on the load record, it is felt that the loads as measured are quite accurate.

TEST RESULTS

Figure 4 shows reproductions of typical oscillograph records for fast and slow loading speeds. The first is for a test which was completed in 1 sec., the other for a 10-min. test. These records show that the time-rate of loading was not uniform, in spite of all attempts to make it so. During the application of the first 15 or 20 per cent of the load, the rate was slow, due probably to deformations in knife-edges, levers and bearing surfaces. The motor speed was held constant throughout each test. After the earlier stages of loading, the load-time record takes a fairly uniform slope, and the rate of loading has been computed from the slope of this straight portion, generally between 25 and 90 per cent of the ultimate load.

The rate of loading was more nearly constant than the rate of strain, as might be expected from the conditions of constant motor speed and the curvilinear stress-strain relation for concrete. Hence rate of loading has been used in discussing the results rather than rate of strain.

Stress-Strain Curves:

By plotting simultaneous readings of stress and strain from records such as those of Fig. 4, the curves of Figs. 5 and 6, for 7 and 28-day tests, respectively, were obtained. Each group of curves for a particular grade of concrete shows a characteristic decrease in both strength and general slope of curve with decrease in rate of loading. While the upper portion of the stress-strain curve is not complete in some cases, the maximum ordinate indicates the approximate strength; the relation of strength and loading speed will be discussed in the following section. While the curves of Figs. 5 and 6 exhibit a gradual decrease in slope, reading from left to right in each group, this relation between slope and speed of loading is shown better in Fig. 7, wherein the secant moduli at 50 and 90 per cent of the maximum load are plotted against the rate of loading. The modulus at 50 per cent maximum load shows a general tendency to increase with the rate of loading; that at 90 per cent maximum load shows a larger and more definite increase as the rate of loading increases. This variation in secant modulus of

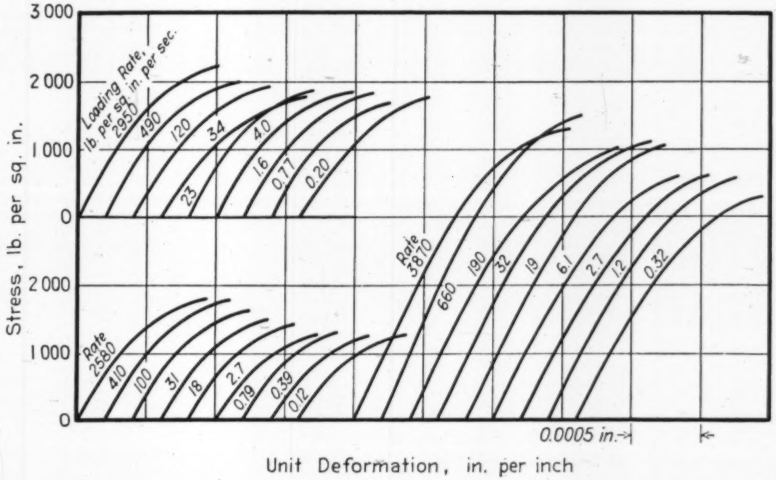

FIG. 5.—Stress-Strain Curves for Concrete Cylinders, 7 days Old.

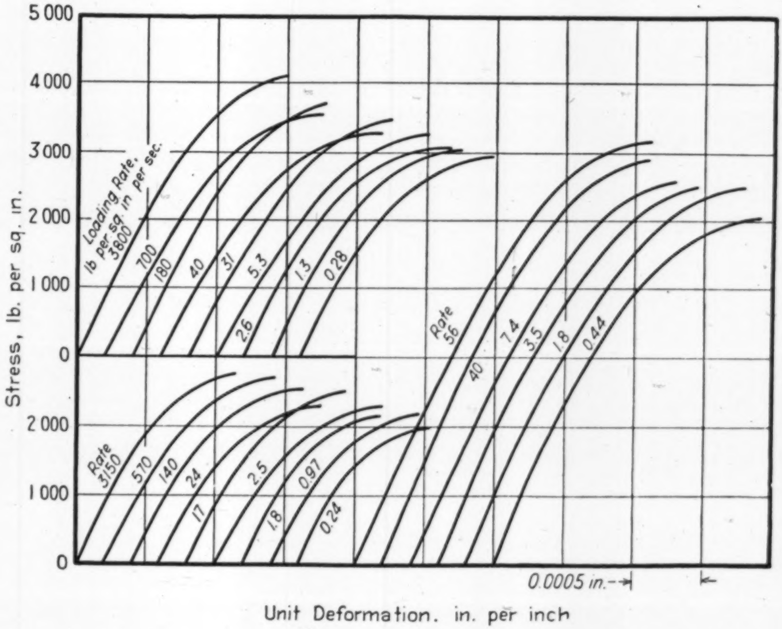

FIG. 6.—Stress-Strain Curves for Concrete Cylinders, 28 days Old.

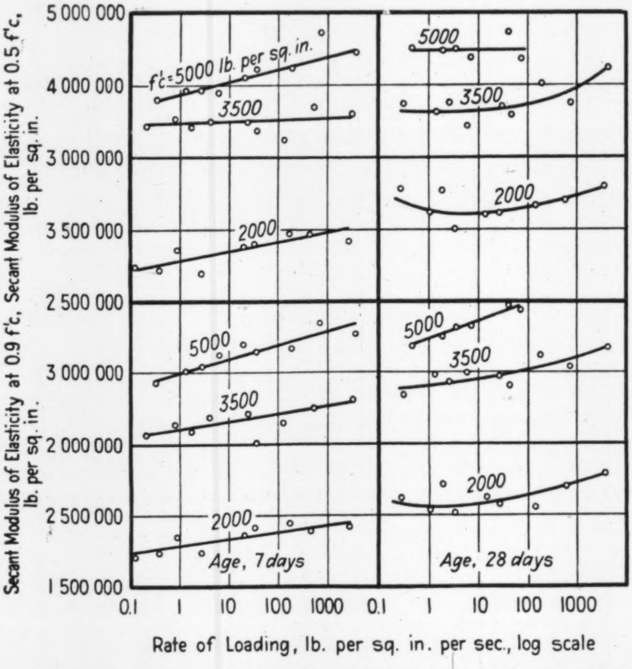

FIG. 7.—Relation Between Secant Modulus of Elasticity and Rate of Loading.

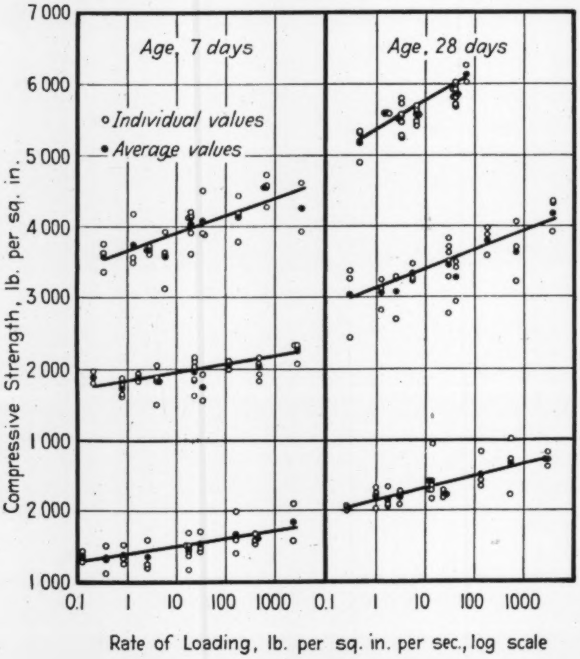

FIG. 8.—Relation Between Compressive Strength and Rate of Loading.

elasticity with testing speed is at least qualitatively in agreement with the results of Glanville's experiments (4). Further comparisons with his tests are discussed in a following section.

Relation Between Strength and Rate of Loading:

Figure 8 presents the relation between compressive strength and rate of loading for three grades of concrete, tested at 7 and 28 days. Values are given both for individual tests and for the average of each group. In this chart, as in Fig. 7, the rate of loading has been plotted on a logarithmic scale. As a result the solid circles indicating average values of strength are represented fairly well by straight-line curves. These straight lines correspond to an equation of the type:

$S = S_1 (1 + k \log_{10} R)$

where S = strength at a given rate of loading R,

S_1 = strength at the rate of loading of 1 lb. per sq. in. per sec., and

k = a constant.

The value for k is roughly 0.07 for the 7-day tests and 0.08 for the 28-day tests. To make the above equation useful it would be desirable to relate it to the nominal testing speeds (in the neighborhood of 30 lb. per sq. in. per sec.) at which the strength of concrete is generally determined. However, in view of the fewness of the test results and the dispersion of the individual values in Fig. 8, it seems unwise to attempt further generalization such as an equation for the relation between nominal strength and loading speed. It is worth noting that a logarithmic relation between strength and speed was also observed in the tests made at the University of Wisconsin (6).

The magnitude of the "speed effect" upon the strength may be emphasized by pointing out for the 28-day tests that the strength for the highest loading rate was as much as one-third greater than that for the lowest rate used in the tests.

Creep of Concrete During Tests:

The difference in the secant moduli of elasticity for concrete tested at various speeds (see Fig. 7) may be considered to be due to plastic deformation or creep of the concrete. This thought formed the basis of a series of tests by Glanville (4), who made observations on concrete cylinders at very small time intervals and drew conclusions which seem to prove his point. He was able to measure strains within 5 sec. after a load was applied, and had to extrapolate values for shorter time intervals. With an oscillograph record of the strains, it is obvious that this difficulty would not exist, and accordingly a few tests were made to verify Glanville's observations. In these tests a concrete cylinder was subjected to a small load, and strains were recorded for a period of 1 min.; an increment of load was added and strains were recorded for another minute, and this was repeated for a number of increments of load. The increase in strain during the minute of constant loading was read from the oscillograph record and the average of such values for three companion cylinders is plotted for each load increment in Fig. 9. The results are similar to those observed by Glanville. The creep at stresses of 175 and 285 lb. per sq. in. are very small; with further increments of load, the creep increases at a rapid rate. At a stress of 885 lb. per sq. in. the creep in 1 min. amounted to 0.00002 in. per inch; at a stress of 1060 lb. per sq. in. the creep was approximately doubled, and at a stress of 1240 lb. per sq. in. the creep amounted to 0.00013 in. per inch in 1 min. This latter stress was 85 per cent of the ultimate strength. Evidently in this test, which lasted about 8 min., the creep amounted to a fairly large part of the total deformation.

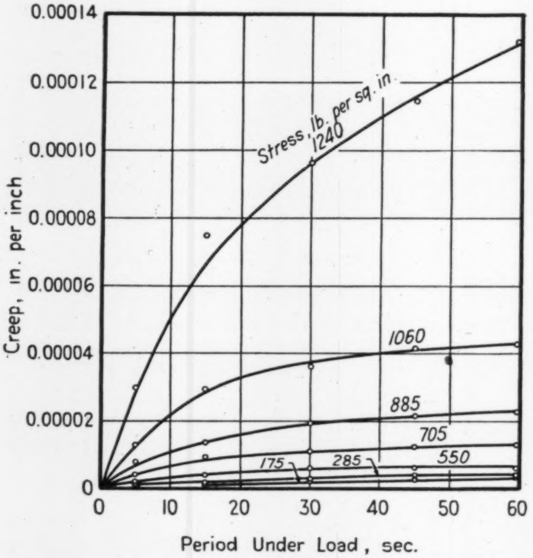

FIG. 9.—Creep of Concrete Under a Stress Applied in Increments.

Age, 7 days. Ultimate strength, 1450 lb. per sq. in.

It is evident that the creep shown in Fig. 9 is not proportional to the applied stress. In this respect the test differs from long-time creep or plastic flow tests, in which proportionality is closely approached. One difference in the two types of test may be noted; whereas the usual test for creep is made on cylinders previously unstressed, the tests recorded in Fig. 9 were made on cylinders in which the cylinder has been subject to previous increments of load. It might be expected that the previous loading would have reduced the capacity for the material to creep and would decrease the ratio of creep to increasing loads. Just the opposite was the case. However, aside from the question of previous loadings, it does seem reasonable that as the stress approaches the ultimate load, the plastic strains should increase more rapidly than the stress.

Another feature of the curves of Fig. 9 is their similarity in shape to creep-time tests in which the time interval is one of months or years rather than seconds. Apparently "time yield" and creep under rapidly applied load are closely allied phenomena, with the main difference in the magnitude of the observed stresses and strains.

CONCLUSIONS

From the foregoing tests, in which compressive stress was applied to 6 by 12-in. concrete cylinders in periods from 1 sec. to 4 hr., the following conclusions have been drawn:

1. A fairly definite relation was observed between the rate of applying load and the strength of the concrete; an increase in rate of loading was accompanied by an increase in strength approximately proportional to the logarithm of the loading rate. The logarithmic relation appears to be incidental and not based on any fundamental law. The ratio of strengths at the highest and lowest loading rates used was as much as $1\frac{1}{3}$ to 1.
2. The secant modulus of elasticity at 50 per cent maximum load showed a tendency to increase with an increase in rate of loading; at 90 per cent of the maximum load there was a consistent increase in the secant modulus with increase in rate of loading. The secant modulus at 50 per cent maximum load did not vary greatly from the initial tangent modulus of elasticity of the concrete.
3. Measurable creep was noted in cylinders loaded for a 1-min. interval. even at load increments as small as 12 per cent of the ultimate strength. With increasing loads, the ratio of creep to load increased rapidly; as the maximum load was approached, the creep formed a very considerable part of the total deformation.

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(4) W. H. Glanville, "Studies in Reinforced Concrete III Creep or Flow of Concrete Under Load," *Technical Paper No. 12*, Building Research Station, England (1930).

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(6) A. J. Klettke, D. W. Webster and F. P. White, "Effect of the Speed of Loading on the Modulus of Rupture of Plain Concrete," Thesis for B.S. Degree, University of Wisconsin (1933). Directed by Prof. M. O. Withey.

DISCUSSION

MR. H. F. MOORE.[1]—The paper by Messrs. Jones and Richart is of marked significance in connection with the work of a section of Committee E-1 on Methods of Testing which is studying the effect of speed of testing on the test results. In the past it has been common to judge of speed of testing by the rate of motion of the moving head of the testing machine. This method has come to be recognized as very unsatisfactory. The relation of speed of head to rate of strain, or to rate of loading, is a very variable one, depending on slip in grips, elastic yield in bearing blocks and elastic yield in parts of the testing machine. Then errors in testing machine load-weighing apparatus become appreciable at high speeds of testing. The three types of testing machine in common use in this country, the balance-beam machine, the machines with pendulum weighing device, and the machines with hydraulic supports and Bourdon gage load indicators, all have different amounts of inertia of weighing mechanism, and machine errors at high speeds would be quite different for the three types. By the use of apparatus such as is described by Jones and Richart the effect of inertia of parts is very small indeed, the telemeters were frequently calibrated during the series of tests, and the results may be taken as showing the variation in properties of the material itself, with a high degree of assurance that errors due to the varying behavior of the testing machine at different speeds have been practically eliminated.

MR. M. O. WITHEY[2] (*presented in written form*).—The data obtained by the authors throw additional light on the relation between speed of loading, strength and elastic properties. It is to be hoped that these investigators will be able to extend their experiments on the strength at still lower rates of stressing approaching the dead load limit. Also a valuable contribution would be the relation of creep to the intensity of the causative stress in mixes of variable cement content. In such a program the creep of the aggregate itself should be determined.

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ELASTIC AND THERMAL EXPANSION PROPERTIES OF CONCRETE AS AFFECTED BY SIMILAR PROPERTIES OF THE AGGREGATE

By L. H. Koenitzer[1]

Synopsis

The elastic properties and thermal coefficient of expansion were determined on specimens cut from the samples of stone, and on concrete specimens containing the crushed stone as coarse aggregate. Tests were made on the stone and concrete specimens in a dry condition, frozen in a dry condition, heated to 190 F., in a moist condition, and frozen in a moist condition. The data presented indicate that engineers should not draw conclusions from the results secured upon individual materials that enter into concrete mixtures, such as the change in volume or the elastic properties of the aggregate, cement, or mortar since a combination of these materials may give an entirely different set of results in the concrete, depending upon the condition of the specimens at the time of testing; that is, if they are in a moist or dry condition, or if frozen or heated to a high temperature. Different combinations of the component parts will, without doubt, produce changes not necessarily in agreement with the properties of the individual parts. The results in the paper are not conclusive but suggest a new field of research work which should prove valuable to users and designers of concrete mixtures.

Introduction

The author in writing the conclusions for his paper presented before the Society last year[2] had his attention called to a similar statement made by Scholer, in a paper on volume changes of concrete presented before the Highway Research Board in December, 1931,[3] which reads as follows:

> "The following condensed outline of those problems which seem to be in the most urgent need of study is presented:
>
> 1. Coarse Aggregates:
>   1. Modulus of elasticity of aggregate.
>   2. Thermal coefficient of expansion of the aggregate.
>   3. Moisture coefficient of expansion of the aggregate.
>   4. The possibility of determining the relationship between these values and the similar values for the concrete in which the aggregate is incorporated.
>   5. Effect of shape of particles upon the above properties, particularly the coefficient of thermal and moisture expansion."

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[1] Assistant Professor, Department of Applied Mechanics, Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

[2] L. H. Koenitzer, "Determination of Modulus of Elasticity and Poisson's Ratio of Concrete at Ages of Fourteen Days to Four Years," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 399 (1935).

[3] C. H. Scholer, "Factors Affecting Volume Changes in Concrete and Their Significance in Design," *Proceedings*, Highway Research Board, Vol. 11, Part I, p. 260 (1932).

Much has been written on aggregates and concrete mixtures, but there has been little or no work done on such a study as suggested by Scholer. The work reported upon in this paper was accordingly undertaken by the Engineering Experiment Station of the Kansas State College. The present paper covers two phases of the subject: namely, the linear change and elastic properties of native stone, neat cement, mortar, and concrete in the following conditions: dry, saturated, frozen, and at a temperature of 190 F.

OUTLINE OF TESTS

Eleven samples of stone were secured from five quarries. In general, each sample of stone consisted of a single piece weighing approximately 150 lb. Each sample represented a different strata of rock. The samples were marked to show the horizontal bedding plane.

Cylinders 2 in. in diameter and 5 in. long were cut out of the blocks of stone parallel and perpendicular to the bedding plane. The remainder of the sample was run through a stone crusher which broke the stone down to a maximum size of $\frac{3}{4}$ in. Three by six-in. cylinders were then made of neat cement, mortar, and concrete containing the above crushed stone as coarse aggregate. The compressive strength, linear change with temperature, and the elastic properties of stone, mortar, neat cement, and concrete specimens were made. The condition of the specimens at the time of test was as follows:

1. Ninety days in moist room, tested moist.
2. Ninety days in moist room, 6 days in laboratory air, tested dry.
3. Ninety-six days in moist room, frozen 24 hr., and tested in moist condition.
4. Ninety days in moist room, 8 days in laboratory air, frozen 24 hr. and tested in dry condition.
5. Ninety days in moist room, 10 days in laboratory air, heated to 190 F. from dry condition, and tested at 190 F.

MATERIALS

Stone:

The stone used in the 100 series of tests was a soft limestone secured near Manhattan, Kans. This stone, secured from the Cottonwood Falls formation, is not considered suitable for pavement work because of low wearing qualitities, although it is very durable, as shown by freezing-and-thawing tests. Samples were selected at random from a number of large pieces from the same quarry.

The stone used in the 200 series was a very durable, hard, gray limestone found in the Plattsburg formation in southeastern Kansas.

The stone used in the 300 series was a durable limestone secured from the Deer Creek formation in southeastern Kansas. It showed considerable wear in the Deval abrasion test.

The stone used in the 500 series was a durable, hard stone secured from the Bethany Falls formation near Kansas City, Mo.

The stone used in the 600 series was a very durable, hard, non-absorbent sandstone from the Dakota formation found in north central Kansas.

The 700 series consisted of mortar specimens.

The 800 series consisted of neat cement specimens.

Sand:

Sand was secured from the Kansas River at Manhattan, Kans. It was a clean, siliceous material having a fineness modulus of 2.83.

Cement:

A commonly used brand of normal portland cement was purchased locally. This cement was tested according to A.S.T.M. Standard Methods C 77 – 32[4] and was found to meet all the standard requirements.

TEST METHODS

Test Specimen:

Stone Cores.—Cores, approximately 5 in. in length, were drilled from the stone samples using a 2-in. core drill with steel shot for an abrasive. Specimens were taken parallel and perpendicular to the bedding plane where possible.

For the determination of the modulus of elasticity in compression, the size of building-stone specimens has been designated by A.S.T.M. Tentative Method C 100 – 31 T[5] as $3\frac{1}{2}$ by $3\frac{1}{2}$ by 12 in. The tentative method specifically requires that the specimens be cut from the original samples with saws: no hand cutting with chisels or other tools is permitted. The author tried using 4-in. cubes cut out with a chisel, but it was found that the specimens were cracked and the outside disturbed by the hammering action, which lowered the modulus of elacity of the specimens. From tests made several years ago by the author[6] on 6-in. concrete cubes cut out on a carborundum saw there was a tendency for the specimens to have a lower compressive strength than molded specimens of the same size. In sawing hard stone the saw heats the surface of the stone regardless of the flow of water on the specimens and this sudden temperature change no doubt damages the surface of the stone which caused the lowering in strength of the specimens. Using a 2-in. steel-shot core drill is not an ideal method for securing specimens, but it appeared to be the best method available at the time, and one which would cause the least disturbance in the surface of the stone. The surface of the core was smoothed off on an emery wheel at the locations where the instrument was to be attached. Great care was used not to drill too fast into the rock, and not to heat the specimens unduly on the emery wheel.

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[4] Standard Methods of Sampling and Testing Portland Cement (C 77 – 32), 1933 Book of A.S.T.M. Standards, Part II, p. 6.

[5] Tentative Method of Test for Modulus of Elasticity of Natural Building Stone (C 100 – 31 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 834 (1931); also 1935 Book of A.S.T.M. Tentative Standards, p. 475.

[6] C. H. Scholer and L. H. Koenitzer, "A Study of Fourteen Brands of Standard Portland Cements and Four Early-Strength Cements," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 581 (1930).

Tests made on 1, 2 and 3-in. cylinders two to three diameters in length showed that there was practically no difference in the results secured on the various size specimens.

Concrete and Neat Cement Cylinders.—After the cores were drilled the remainder of the stone sample was crushed in a rock crusher. The crushed stone was then separated into two sizes: material passing the $\frac{3}{4}$-in. and retained on the $\frac{3}{8}$-in. sieve, and material passing the $\frac{3}{8}$-in. and retained on the No. 16 sieve. The materials were then weighed out in the following proportions for each batch:

$\frac{3}{4}$ to $\frac{3}{8}$-in. stone	6500 g.
$\frac{3}{8}$ to No. 16 stone	6500 g.
Sand	6500 g.
Cement	4500 g.
Water-cement ratio by volume	0.8

The concrete test specimens were made in accordance with A.S.T.M. Methods C 39 – 33,[7] except a departure from this method was made in using 3 by 6-in. compression specimens for concrete containing a maximum size aggregate of $\frac{3}{4}$ in. The materials were mixed together dry, and then the water was added. An experienced operator did all of the mixing and placing of the concrete in the 3 by 6-in. cylinder molds. The concrete was mixed by hand for approximately 3 min. A slump test was made (A.S.T.M. Method D 138 – 32 T).[8] The concrete was placed in 3 by 6-in. steel molds and cured according to A.S.T.M. Methods C 31 – 33.[9]

Two stainless-steel plugs $\frac{1}{2}$ in. in length are placed in the ends of each specimen to be tested for linear change.

For the neat cement specimens, sufficient water was added to get a slump of 2 in., which was the average slump of the concrete mixtures.

Curing.—All concrete and neat cement specimens were cured in the moist room 90 days before testing.

Condition of Specimens.—Specimens noted as moist were taken directly from the moist room and tested either at laboratory temperature (70 to 80 F.) or in a frozen condition. Specimens noted as wet had been soaked in water to constant weight and tested in laboratory air as mentioned above. Specimens noted as dry were dried out in laboratory air to approximately a constant weight and tested at laboratory temperature. The specimens tested in a frozen condition were placed in the freezer directly from the moist room or the laboratory air. The specimens were removed from the freezer

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[7] Standard Methods of Making Compression Tests of Concrete (C 39 – 33), 1933 Book of A.S.T.M. Standards, Part, II, p. 230.

[8] Tentative Method of Test for Consistency of Portland-Cement Concrete (D 138 – 32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 775 (1932); also 1935 Book of A.S.T.M. Tentative Standards, p. 841.

[9] Standard Methods of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 33), 1933 Book of A.S.T.M. Standards, Part II, p. 225.

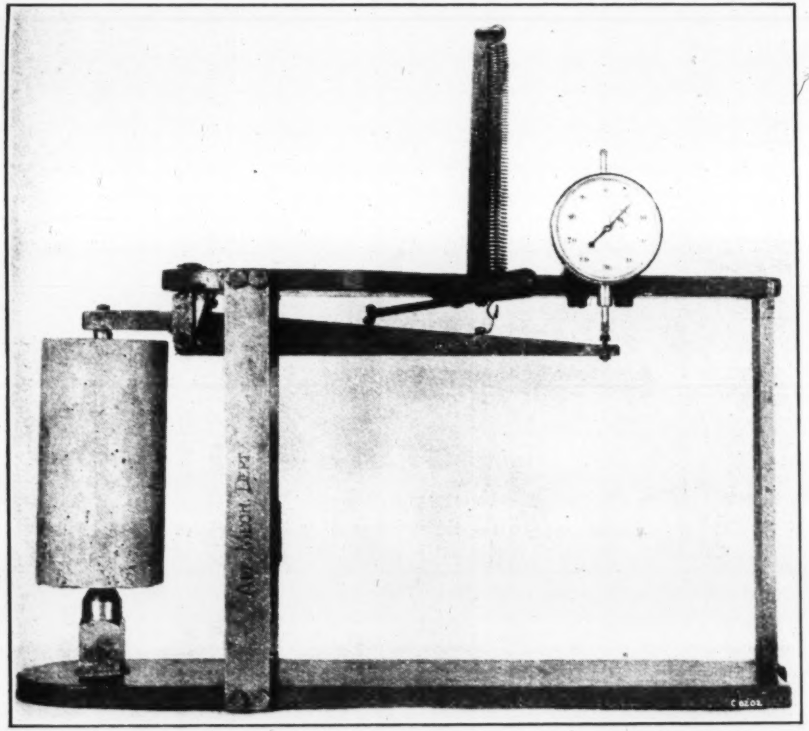

Fig. 1.—Apparatus for Measuring the Linear Change of Specimens.

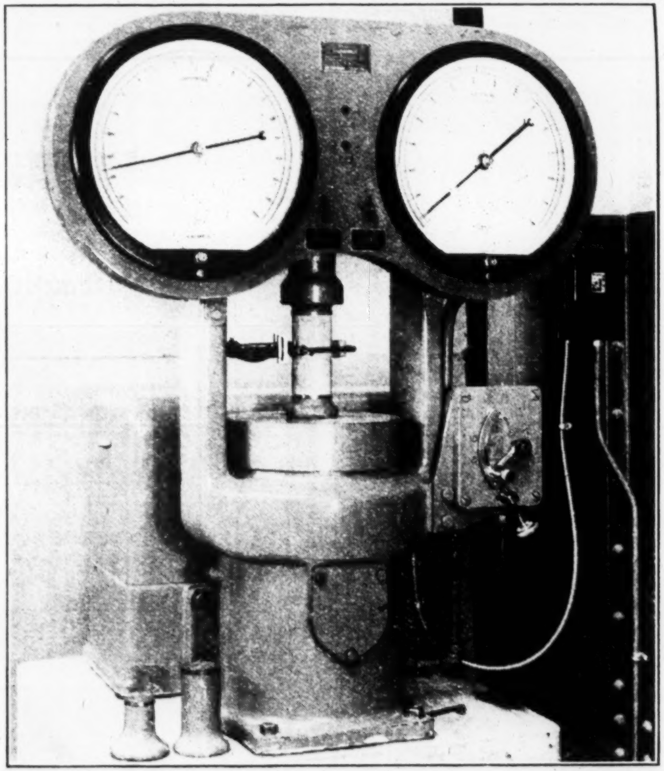

Fig. 2.—Specimens Being Tested with Huggenberger Tensometer Attached.

24 hr. later at a temperature of approximately 9 F. and tested immediately. The specimens remained frozen throughout the test as evidenced by frost remaining on the surface of the specimen during the test.

Freezing.—Specimens were placed for 24 hr. in a mechanical refrigeration box made especially for the freezing of concrete specimens and aggregates. The box was tested showing that 3-in. cylinders were frozen solid in less than 3 hr. The temperature of the freezing chamber before the removal of the specimens was 9 F.

TABLE I.—COEFFICIENTS OF LINEAR EXPANSION.

Change × 10^{-6} per Unit Length per deg. Fahr.

Series	Moist Condition			Dry Condition		
	Frozen 9 to 80 F.	Heated 83 to 190 F.	Change 9 to 190 F.	Frozen 9 to 80 F.	Heated 83 to 190 F.	Change 9 to 190 F.
CONCRETE SPECIMENS						
100	5.88	5.85	6.17	4.44	4.18	4.61
110	6.26	5.07	5.93	5.01	4.50	4.91
120	6.23	5.60	5.88	4.13	5.64	5.39
130	5.92	5.21	5.73	4.38	4.39	4.71
140	5.64	5.47	5.42	4.50	5.75	5.61
210	3.77	4.90	4.88	3.79	3.58	4.14
220	4.13	4.68	4.66	4.96	4.65	5.12
230	6.19	5.78	6.32	5.07	7.08	6.90
310	5.10	5.80	5.59	5.62	5.45	6.27
320	3.99	4.98	5.31	4.30	4.67	5.74
330	4.96	4.87	4.96	4.48	5.71	5.81
510	4.44	5.13	4.49	4.14	4.66	4.66
520	4.82	4.78	5.13	3.86	5.24	5.08
530	4.87	4.94	4.84	4.76	4.37	4.85
610	7.06	7.89	7.37	6.01	4.46	5.46
620	6.86	7.14	7.08	6.30	4.90	5.60
630	7.07	7.15	7.04	6.23	7.91	7.76
700[a]	7.66	6.71	7.06	7.12	7.12	8.18
800[b]	11.80	7.82	8.45	12.10	8.72	11.80
STONE SPECIMENS PERPENDICULAR TO THE BEDDING PLANE						
110	3.83	1.75	3.00	......	......	......
210	......	......	......	3.35	2.31	2.86
220	......	......	......	3.22	3.50	2.59
230	......	......	......	6.14	5.30	5.70
310	......	......	......	3.61	4.42	4.52
330	......	......	......	4.32	5.55	5.43
400	......	......	......	3.46	4.02	3.89
620	......	6.43	......	3.42	5.22	5.68
STONE SPECIMENS PARALLEL TO THE BEDDING PLANE						
100	3.89	3.25	3.61	......	......	......
510	2.68	2.15	2.52	......	......	......
530	2.42	2.91	3.00	......	......	......
620	5.44	5.22	5.67	5.67	7.38	7.72

[a] Mortar.
[b] Neat cement.

Heating.—The specimens were placed in a large electric drying oven maintained at 190 F. by a thermostatically controlled electric unit.

Apparatus:

Linear Change with Temperature.—The apparatus used for measuring the linear change of the specimens is shown in Fig. 1. The temperature of the room was noted so that the standard invar steel reference bar could be corrected to standard conditions. The Federal dial on the apparatus is graduated to 0.0001 in., and the leverage is 5 to 1, making each division on the dial 0.00002 in. The linear change is shown in Table I for the various conditions and exposures of the specimens.

TABLE II.—MODULUS OF ELASTICITY.
Condition of Specimens at Time of Test.

Series	Modulus of Elasticity, lb. per sq. in.					
	Wet	Moist	Frozen Moist	Dry	Frozen Dry	Heated to 190 F.
			CONCRETE SPECIMENS			
100	3 345 000	3 160 000	4 401 000	4 040 000	3 730 000	3 232 000
110	......	4 215 000	3 875 000	3 975 000	......	3 345 000
120	4 401 000	4 040 000	3 903 000	3 672 000	3 590 000	......
130	......	3 160 000	3 470 000	4 085 000	3 875 000	......
140	3 160 000	4 401 000	4 085 000	3 467 000	3 875 000	......
210	4 845 000	3 727 000	4 887 000	5 105 000	......	......
220	5 390 000	4 620 000	4 845 000	4 845 000	5 105 000	......
230	4 401 000	4 215 000	5 105 000	5 390 000	5 390 000	3 875 000
310	4 620 000	4 510 000	5 003 000	4 398 000	3 875 000	3 232 000
320	4 085 000	......	......	6 250 000	......	......
330	......	3 470 000	4 085 000	4 117 000	4 215 000	3 062 000
510	5 105 000	4 620 000	5 390 000	......	......	......
520	3 902 000	3 875 000	4 620 000	4 401 000	......	3 590 000
530	4 845 000	4 215 000	4 845 000	......	......	......
610	3 730 000	3 232 000	6 250 000	5 190 000	5 105 000	4 085 000
620	......	5 705 000	5 705 000	6 017 000	4 215 000	......
630	......	......	......	5 347 000	5 705 000	3 062 000
640	4 845 000	6 250 000	5 390 000	4 737 000	5 105 000	......
650	......	4 215 000	6 250 000	......	......	......
700	......	3 450 000	3 870 000	3 875 000	......	2 852 000
800	4 401 000	3 990 000	3 470 000	2 852 000	3 470 000	1 385 000
			STONE SPECIMENS PERPENDICULAR TO BEDDING PLANE			
100	4 215 000	3 875 000	10 775 000	4 845 000	......	......
110	3 470 000	4 215 000	10 775 000	4 215 000	......	......
210	6 250 000	5 105 000	13 855 000	7 455 000	......	6 250 000
220	8 080 000	6 930 000	10 775 000	12 120 000	......	......
310	4 085 000	6 465 000	8 815 000	5 705 000	......	......
320	6 250 000	6 465 000	8 080 000	7 455 000	......	......
330	......	6 930 000	12 120 000	7 455 000	......	5 705 000
510	5 105 000	4 401 000	24 240 000	6 250 000	......	......
520	4 085 000	3 730 000	4 215 000	3 875 000	5 390 000	......
530	5 105 000	1 204 000	......	5 705 000	......	......
620	7 400 000	8 000 000	16 160 000	9 800 000	......	......
			STONE SPECIMENS PARALLEL TO BEDDING PLANE			
110	1 864 000	......	......	2 694 000	......	......
520	2 694 000	......	......	3 875 000	......	......
530	5 705 000	......	......	6 930 000	......	......
620	9 710 000	8 080 000	7 455 000	10 775 000	......	......

Elastic Measurement.—The stone and concrete cylinders were capped on the ends with neat cement paste. The specimens were then placed in a Baldwin-Southwark 75,000-lb. hydraulic testing machine, as shown in Fig. 2. The machine is equipped with a speed indicator showing the rate of applications of the load. The load was applied to the specimens at the rate of 570 lb. per sq. in. per min. A maximum load of 1500 lb. per sq. in. was applied.

TEST DATA

Modulus of Elasticity:

In determining the modulus of elasticity of stone and concrete specimens, a Huggenberger Tensometer with a 1-in. gage length was placed at the center of the specimen. The multiplication ratio for the Tensometer was 1212, which enabled the operator to read a deformation of 0.00004125 in.

The results shown in Table II are based upon tests made on one or more specimens. Three sets of readings were taken on each specimen, each set being 120 deg. apart on the circumference of the specimen and the average of the three sets of readings recorded. In most cases the compressive and elastic properties and linear change were practically the same for specimens of the same series.

It will be noted in Table II, that the results for the 100 series, representing stone taken at random, are not uniform, which is substantiated later in this report for tests made on stone from the same formation but from different strata. Tests made on stone from the same stratum give fairly close results.

Figure 3 shows that for stone specimen No. 1003 the modulus of elasticity of the specimen frozen moist was over twice as high as when the specimen was tested at room temperature in a moist, wet or dry condition. The concrete specimens showed less variation in the elastic properties for the various conditions at the time of test.

The 200 series, in which durable gray limestone was used, showed a slightly higher modulus of elasticity than did the soft limestone in the 100 series. From the data presented in Table II it appears that the stone specimens from the center strata, 220 series, were more rigid than the specimens from the strata above and below the center, when the stone was in a moist or saturated condition. The concrete specimens containing this stone showed a slightly higher modulus of elasticity when moist and wet than did the specimens containing stone from the other two strata. Figure 4 shows a closer grouping of the elastic properties of the 220 series, both for the stone and concrete specimens when tested under the various controlled conditions.

The 300 series showed no outstanding qualities or characteristics in so far as the elastic properties of the stone or concrete specimens were concerned. The series is not complete, as shown in Table II.

In the 500 series the center stratum had a lower modulus of elasticity than did the strata above and below the center. This is opposite to the condition found in the 200 series.

The five samples of sandstone in the 600 series recorded in Table II show that the concrete specimens containing the sandstone had, on the

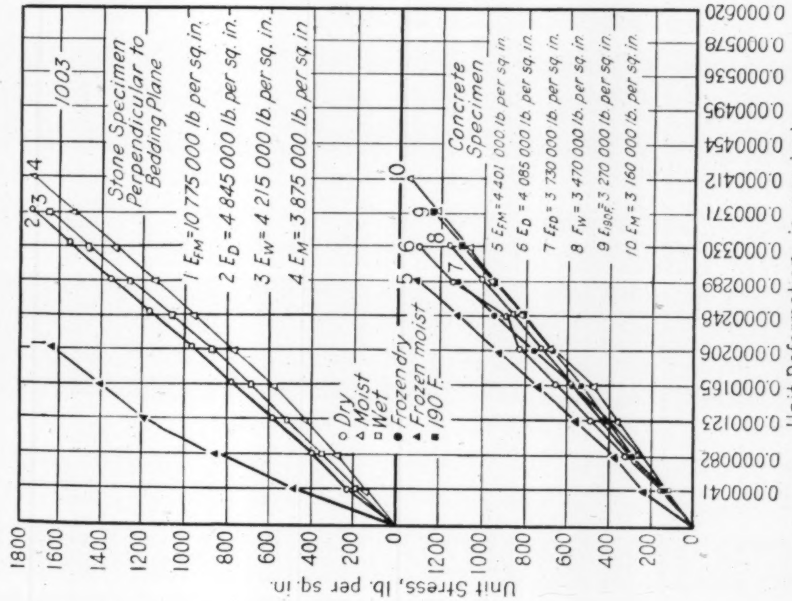

FIG. 3.—Modulus of Elasticity of Stone and Concrete Specimens Under Various Conditions (100 Series).

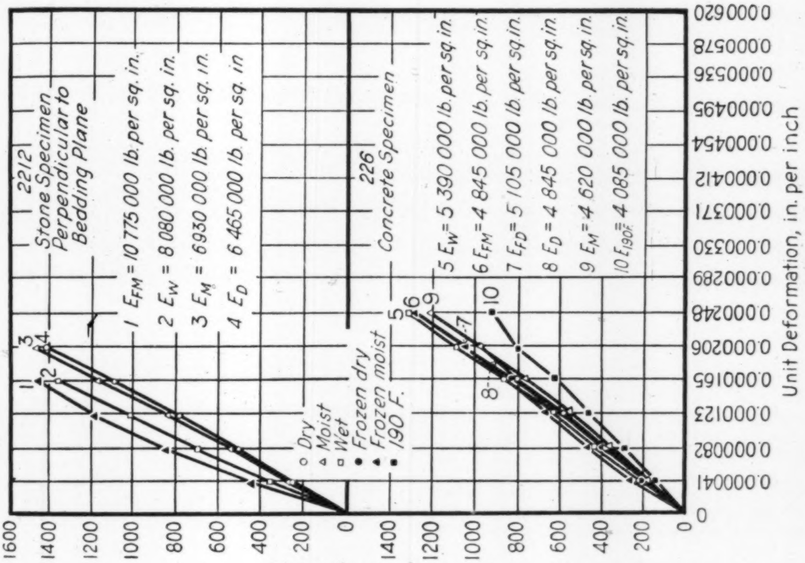

FIG. 4.—Modulus of Elasticity of Stone and Concrete Specimens Under Various Conditions (200 Series).

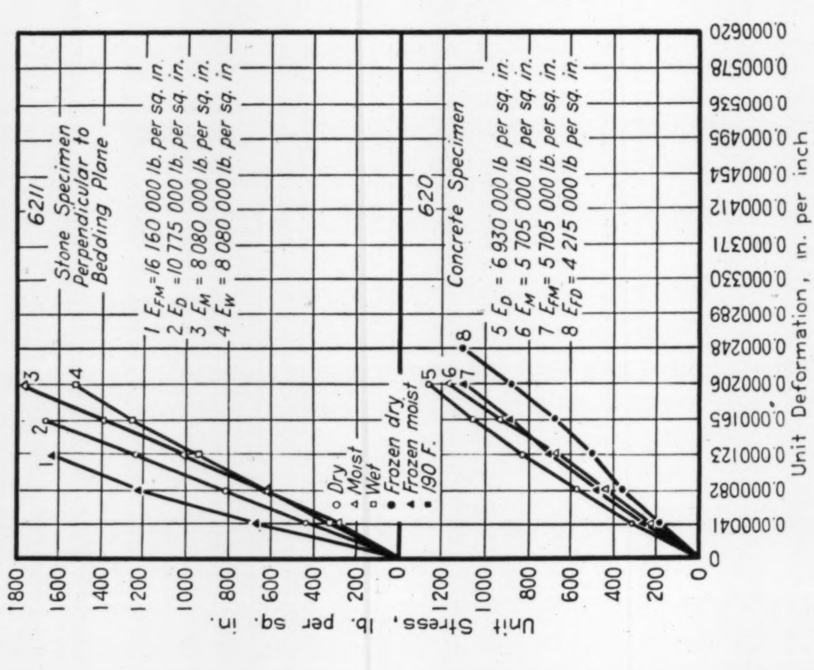

FIG. 5.—Modulus of Elasticity of Stone and Concrete Specimens Under Various Conditions (600 Series).

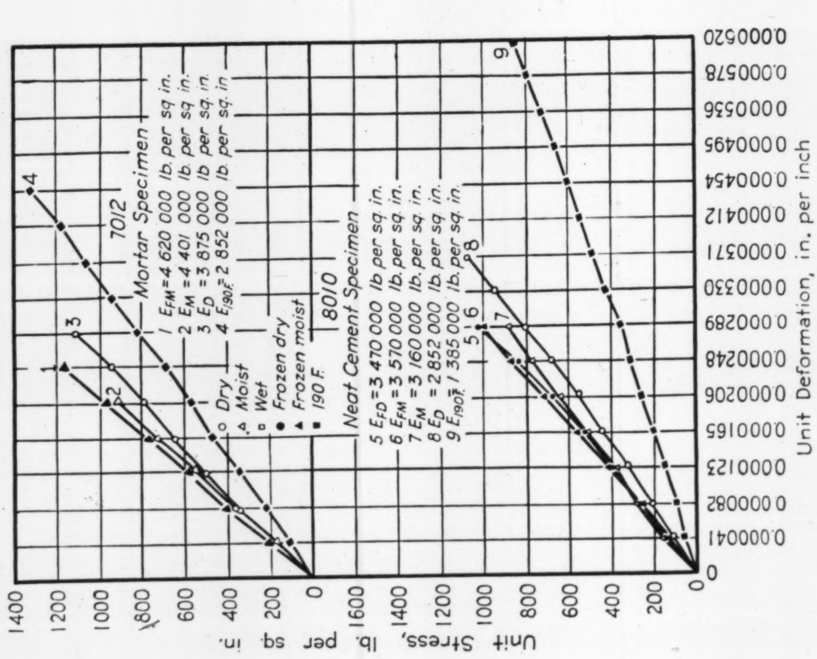

FIG. 6.—Modulus of Elasticity of Mortar and Neat Cement Specimens Under Various Conditions.

whole, a higher elastic modulus than did the concrete containing limestone. Figure 5 shows the curves for a representative sandstone.

The mortar specimens in the 700 series showed very low elastic moduli when compared either with stone or with concrete specimens. The mortar specimens taken from the moist room had an average modulus of elasticity of 3,450,000 lb. per sq. in. when tested in a moist condition; 3,870,000 lb. per sq. in. when frozen moist; 3,875,000 lb. per sq. in. when tested dry at laboratory temperature, and 2,852,000 lb. per sq. in. when heated to 190 F.

The neat cement specimens, 800 series, on the whole showed lower moduli of elasticity than those of the stone, concrete, or mortar specimens.

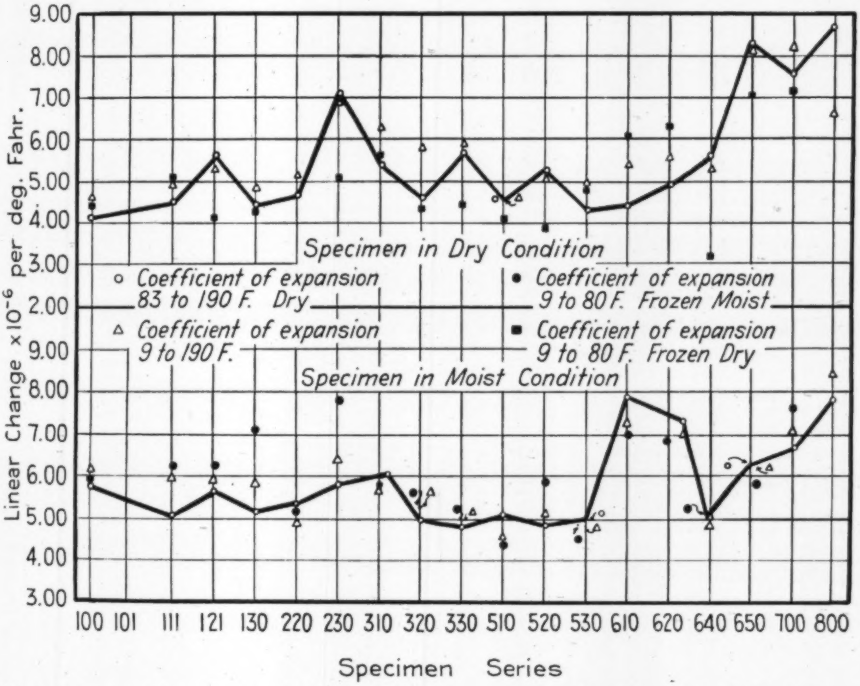

FIG. 7.—Thermal Coefficients of Linear Expansions for Concrete, Mortar, and Neat Cement Specimens Under Various Conditions.

The drier the neat cement specimens when tested, the lower the modulus of elasticity. At a temperature of 190 F. the modulus was 1,385,000 lb. per sq. in., which was the lowest value found. The results are plotted in Fig. 6.

Linear Change with Temperature of Concrete and Stone Specimens:

Table I shows the computed coefficients of linear expansion for concrete and stone specimens under various conditions. The calculations in the table are based upon the change in length, due to the special treatment, from that of the original condition; that is, the lengths of specimens as taken from the moist room, or dried in the laboratory air.

It will be noted in Table I and Fig. 7 that the concrete made with the soft limestone in the 100 series showed a considerably lower coefficient of expansion when the specimens were dry than when they were moist. This was true regardless of the temperature range.

For the 300 series there was a tendency for the opposite to be true. The dry specimens showed a greater change in length than did the moist specimens for both temperature ranges.

TABLE III.—COMPRESSIVE STRENGTH OF CONCRETE SPECIMENS UNDER VARIOUS CONDITIONS AT TIME OF TEST.

Series	Compressive Strength, lb. per sq. in.				
	Moist	Frozen Moist	Dry	Frozen Dry	Heated to 190 F.
100	5848	7 852	6 384	...	2829
110	5588	7 878	5 627	...	3500
120	4916	7 025	5 546	...	3126
130	5426	8 262	6 663	...	3126
140	5871	9 068	6 168	6 154	...
210	7314	10 054	8 206	9 168	...
220	6139	9 100	4 605	5 350	4004
230	6253	9 341	6 131	6 614	...
310	6437	...	6 161	5 121	3586
320	6630	9 170	5 442	6 875	...
330	...	9 539	4 919	5 532	...
510	5696	7 313	5 638	6 310	4958
520	6415	9 323	3 242	6 359	...
530	5911	...	4 937	4 688	2758
610	6184	7 168	5 777	6 667	3780
620	...	8 051	6 493	6 741	...
630	5311	5 546	6 027	5 744	3926
640	5850	7 993	6 401	5 913	4386
650	...	6 335	6 432	6 076	4371
700[a]	3537	...	5 022	5 696	...
800[b]	4669	...	10 635	10 887	3883

[a] Mortar.
[b] Neat cement.

The concrete containing the sandstone of the 600 series showed greater coefficients of expansion in both moist and dry conditions than did any of the concrete using the limestone aggregate. The sandstone specimens themselves have a coefficient of expansion somewhat higher than the limestones; however, the greatest change occurs when the aggregate is included as part of the concrete mixture. The mortar specimens showed practically as great a change in length as did the concrete containing the sandstone aggregate. The mortar specimens tested dry showed the greater change. The neat cement specimens in turn showed a greater change in length than did the mortar specimens for the various conditions, the specimens tested dry showing the greatest change per unit length.

Compressive Strength of Concrete Specimens:

In Table III are shown the results of the compression tests made on the specimens after they had been used for determining the elastic and linear properties of the concrete. The 100 series concrete specimens showed a higher compressive strength when tested in a dry condition than when tested moist. This is to be expected as the aggregate, being a soft limestone, has an absorption of over 4 per cent, which for this particular material seems to decrease the strength of the resulting concrete. In most of the concrete specimens in the other limestone series, the moist specimens showed a slightly higher strength than did the dry specimens.

The mortar specimens showed a lower strength than did the concrete specimens containing the coarse aggregate. The neat cement specimens showed practically 100 per cent higher strength than did the mortar specimens. This is perhaps due to the lower water-cement ratio for the neat cement specimens. The strength of the dry neat cement specimens was the same as for the specimens frozen dry.

Heating to 190 F. considerably decreased the compressive strengths of all specimens.

SUMMARY AND CONCLUSIONS

1. Each of the ingredients that enters into a concrete mixture plays an important but somewhat unknown part in fixing the volumetric and elastic properties of the resulting concrete.

2. The elastic properties and linear change with temperature of the individual materials entering into the concrete mixture are not indicative of the results secured with the concrete mixture.

3. The elastic and thermal expansion properties of the different materials vary among themselves. For any one material those properties vary with the condition of test, the greatest variation being caused by freezing the specimen.

4. The elastic and linear properties of stone from different strata in the same quarry may be different. Hence, in making further studies it is suggested that only the main strata of stone be used from quarries in different geological formations. This would reduce the number of test samples required.

5. The results in this paper are not conclusive but perhaps point the direction toward which a detailed study should be made. Considerable work is involved in the making of the specimens, securing the data, and summarizing the results. Much of the data is included in this report without any discussion, for the benefit of those who might wish to carry through a

similar project. No doubt many states would have materials that would give valuable information if such a study were made.

6. For further study, it is suggested that the linear change of the concrete specimens be based upon the lengths of the specimens when they are 24 hr. old, in a moist condition.

7. It is also suggested that a definite study be made on stone specimens to determine the coefficient of expansion due to change in moisture content. Studies made on concrete specimens have shown that concrete grows, due to changes in temperature and moisture content.

DISCUSSION

MR. H. J. GILKEY[1] (*by letter*).—As the author stated, many of the results of the investigation are necessarily inconclusive and doubtless the greatest immediate contribution is the blazing of a trail into new territory, continued exploration of which promises to contribute much to a better understanding of concrete in its relationship to its constituent parts. Two of the purported findings are so squarely at variance with some of the writer's observations that have been checked so many times and under such a variety of conditions that he deems it desirable at least to flag them as questionable.

Table III is cited as authority for the statement that the compressive strengths were higher for the moist-tested specimens than for those tested dry except for series 100. From a study of Table III the writer believes that the most that can be said is that the data are inconclusive upon this point; the indications of series 200 are about evenly divided; series 300 and 500 support the statement but with so great a range of strengths that question is raised whether or not some of the specimens may not have been faulty or the tests questionable. The 600 series is virtually in agreement with series 100. The mortars of the 700 series are in good agreement with the writer's own observations which include both mortars and concretes of a variety of aggregates, proportions, ages, and water-cement ratios. He has found no difference in the effect of air drying upon the strengths of mortars and concretes.

The effect of air drying for series 800 is very pronounced but the writer has not investigated the subject for neat cement and is not prepared to say that this indication is abnormal, although he suspects that it may be.

Upon exposure to air at ordinary temperatures following a period of moist curing, a substantial increase in the strength occurs, the maximum being attained after from 10 days to 3 or 4 weeks, depending upon the relative humidity of the air, the size of specimen, and possibly somewhat upon the mixture. While the humidity of the air was not indicated, the 3 by 6-in. specimens of these tests should probably have acquired from one-half to two-thirds of the increase available by air drying during the exposure to which they were subjected. The increase seems to be solely due to drying, as is demonstrated by the fact that re-immersion for one day just prior to test reduces the strength to that which it had when first exposed. In fact, after the maximum strength is attained, continued drying often produces a slow partial reduction in strength and after several months' exposure the dry tested strength may be below the maximum attained and the re-soaked wet-tested strength correspondingly below the strength when the drying was started. The accompanying Table I gives results from some very recent unpublished tests performed by the writer under the auspices of the Iowa Engineering Experiment Station.

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The substantial increase in the strength of specimens subject to only a few days' drying indicates that the period of exposure that was used in the author's tests was probably sufficient to increase the strengths materially even though the maximum was probably not attained.

TABLE I.—EFFECT OF AIR DRYING FOR VARIOUS PERIODS UPON COMPRESSIVE STRENGTHS OF CONCRETE.

Each result is the average of two tests.

Age, days		Compressive Strength, lb. per sq. in.			Ratio to Initial Strength		Ratio of Dry Strength to Resoaked Strength
Until Time of Exposure to Air	At Test	Initial	Air Dry	Resoaked	Air Dry	Resoaked	
28	84	4700	6670	4790	1.42	1.02	1.40
56	84	5610	7210	5490	1.29	0.98	1.32
63	84	....	7360	5655	....	....	1.30
70	84	....	7510	6110	....	....	1.23
77	84	6050[a]	7500	6040	1.24	1.00	1.24
81[b]	84	6050	7090	6275	1.17	1.04	1.13
28	180	4700	7020	4800	1.50	1.02	1.46
84	180	6050	8140	5490	1.35	0.91	1.48
28	365	4700	6925	4900	1.47	1.05	1.41
56	365	5610	7610	5375	1.36	0.96	1.42
84	365	6050	8160	5500	1.35	0.91	1.49
180	365	6900	7900	5710	1.15	0.83	1.38
280	365	6975	7940	6150	1.14	0.88	1.29
308	365	6930	8140	6140	1.18	0.89	1.33
337	365	6780	8500	6825	1.25	1.01	1.25
344	365	7060[c]	8475	6840	1.20	0.97	1.24
351	365	7060	8600	7112	1.22	1.01	1.21
358	365	7060	8525	....	1.21	....	....
362[b]	365	7060	7825	....	1.11	....	....
363[b]	365	7060	7465	....	1.06	....	....

[a] 84-day strength.
[b] Short exposure, maximum strength not attained.
[c] 1-yr. standard strength.

The results of other tests showing the effects of air drying on strength have been published in the following papers:

H. J. Gilkey, "Effect of Varied Curing Conditions upon Compressive Strength of Mortars and Concretes," *Proceedings*, Am. Concrete Institute, Vol. XXII, p. 424 (1926).

H. J. Gilkey, Discussion on "Finishing and Curing of Concrete Roads," *Transactions*, Am. Soc. Civil Engrs., Vol. 91, pp. 161–165 (1927).

J. L. Savage, I. E. Houk, H. J. Gilkey, and F. Vogt, "Arch Dam Investigation," Vol. II, pp. 438–445, 460–465, The Engineering Foundation, New York City (1934).

H. J. Gilkey, Discussion on "Experiments with Concrete in Torsion," *Transactions*, Am. Soc. Civil Engrs., Vol. 100, pp. 972–973 (1935).

The writer has data from several other unpublished series which supply more evidence of the same kind.

The other question is regarding the apparent indication that the concrete specimens were stronger than the mortar specimens. Table III clearly supports the statement but in many series of tests the writer has yet to experience a case in which a mortar did not have a strength substantially above that of its concrete of equal water-cement ratio.

A discussion presented by the writer before the Society in 1930[2] supplies some representative comparisons of mortars and their concretes for several mixtures and water-cement ratios at two test ages. Other tests comparing the strengths of concretes with their mortars for a variety of proportions, gradings, and water-cement ratios are also included.

TABLE II.—COMPARISONS OF CONCRETES WITH THEIR MORTAR.

Coarse Aggregate	Condition of Coarse Aggregate			Ratio, Strength of Mortar to Strength of Concrete		
	Dry	Soak 15 min.	Saturated	Dry	Soak 15 min.	Saturated
Crushed Limestone (A)	3120	2500	2185	1.10	1.37	1.57
Crushed Limestone (B)	3255	2410	2310	1.05	1.42	1.49
Crushed Limestone (C)	2950	2485	2290	1.16	1.38	1.50
Crushed Limestone (Bedford)	1180[a]	2025	1950	2.90[a]	1.69	1.76
Gravel (A)	3130	2210	2230	1.09	1.55	1.53
Gravel (B)	3290	2540	2240	1.04	1.35	1.41
Gravel (C)	2920	2000	2120	1.18	1.72	1.62
Crushed Granite (A)	2810	....	2175	1.22	....	1.58
Crushed Granite (B)	2830	....	2060	1.21	....	1.67
Crushed Sandstone (A)	1150[a]	1785	1620	2.99[a]	1.93	2.11
Crushed Sandstone (B)	1810[a]	....	2090	1.89[a]	....	1.64
Broken Stove Iron	1870	....	2190	1.83	....	1.57
None (Mortar)	3430	....	....	1.00	1.00	1.00

[a] Very absorbent coarse aggregate made mix too dry for proper placement.

REMARKS.—Equal absolute volumes of coarse aggregate used in all batches. The mortar (cement, sand and water) was identical for all batches. The actual water-cement ratio was below the nominal in all cases where the aggregate was not pre-saturated. Note the great increase in strength in left-hand column due to lowering of water-cement ratio by absorption which invalids any comparison of mortar *versus* concrete for these specimens. The sand was always dry when added to the batch, both for concretes and mortar. Each concrete strength is the average 28-day strength of four specimens cast on different days and standard cured. The mortar strength is the average for 25 specimens several of which were cast on each casting date.

The writer has many additional unpublished data, all of which show the mortar unquestionably to have the greater strength. In one instance 25 mortar specimens were cast as controls for a rather extensive series of tests comparing concretes made from a variety of representative coarse aggregates. A few of the results are given in the accompanying Table II. Even for the limestones and gravels which are recognized as the best of aggregates the mortar strengths overun by from 40 to 60 per cent.

If it so happens that in the author's tests the coarse aggregate was introduced to the mixture dry without the addition of sufficient water to compensate for absorption, the indication of Table III might be explained on that basis but under those conditions it is not valid to state that the concrete is stronger than its mortar, for the water-cement ratio of the concrete would have been below that of its mortar.

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[2] H. J. Gilkey, Discussion on Effect of Size of Aggregate on Concrete, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 524 (1930).

Such details as the specific gravities and absorptions of the several stones and the numbers of specimens represented by each strength result of Table III and whether or not the range of variations in the strengths of individual specimens was as great as it is in some cases for the group averages, would all be helpful in promoting a better understanding of the project.

MR. L. H. KOENITZER[3] (*author's closure by letter*).—Mr. Gilkey's discussion centers about secondary conclusions in the body of the paper and does not in any way refer to the major conclusions listed in the summary. The author believes that the evidence as shown in the data submitted was sufficient to justify commenting upon the facts but did not consider the data sufficient to warrant drawing any final conclusions.

Data as to the water-cement ratio used and the condition of the materials is shown on page 396. It will be noted that absorption of water by the stone could not have been a contributing cause to the higher strengths shown by the concrete as compared with the mortar.

Although it is not clear from Mr. Gilkey's data just what the mixes are in his Table I, it is believed that the mortar he refers to is a similar mortar to that used in the concrete shown. This would give for the mortar specimens a very high cement content and high slump compared to the author's mortar specimens in which fine aggregate was added until the same consistency was secured as in the concrete specimens. It is believed that this might easily account for the differences as observed by Mr. Gilkey.

The presentation of adverse data does not necessarily disprove previous data or theories, but perhaps emphasizes the fact that the results in different parts of the country may be based upon different conditions and materials. If systematic nation-wide cooperation was received in the studying of aggregates and concrete mixes as is being made on cement at the present time, it is the author's opinion that there would be far greater progress made in the concrete field and perhaps we would come nearer to solving our durability and volume change problems.

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THE CONE METHOD FOR DETERMINING THE ABSORPTION BY SAND

By D. O. Woolf[1]

Synopsis

This paper discusses the methods which have been used for determining the absorption by sand and indicates the difficulties and errors inherent in these methods. The development of the cone method is briefly stated and the method is presented in detail. The results of a cooperative series of tests, performed by twelve laboratories, in which the cone method is compared with three other absorption methods, are presented. The conclusion reached by this series of tests is that the cone method is superior to the other methods used and that the cone method furnishes a reliable and accurate result with a minimum of equipment and working time.

Ever since the design of concrete mixtures was approached from a scientific viewpoint, there has been a need for an accurate method of determining the absorption of water by the aggregates. Only a portion of the water used in mixing concrete is needed for the complete hydration of the cement. The rest is necessary to lubricate the mix so that it may be placed uniformly and without undue difficulty. Some of this lubricating water may be absorbed by the aggregates, but the remainder, the so-called "free" water, dilutes and weakens the cement paste. Consideration of the strength of the resulting concrete must involve the amount of this diluting water, and a knowledge of the water absorbed by the aggregates is necessary to determine the net water content available for the cement. The determination of the bulk specific gravity and from this the bulk volume and weight of the aggregates required for a given yield of concrete also necessitates a determination of the amount of absorbed water.

The question of determining the water absorption of fine aggregate by means of a simple yet reasonably accurate test has been given considerable attention during the past several years and a number of methods have been devised. The majority of the procedures so far suggested are based on the observed behavior of sand grains in the presence of free moisture. Extensive study by the U. S. Bureau of Public Roads has disclosed that most of these methods are liable to furnish inaccurate results. Some require great care in the test manipulations and are so sensitive to nominally uncontrolled variables that their use in routine testing is not warranted. The various procedures which have been suggested are briefly reviewed. The so-called "cone method" developed by the author after considerable study of this problem is then discussed.

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Probably the first method used for determining the condition usually known as "saturated and surface-dry," when the permeable pore spaces in the sand grains are filled with water without any moisture adhering to the surfaces of the grains, was by simple visual inspection. The procedure is simply to spread the sand out on a smooth surface and permit it to air dry, with or without artificial circulation. The sample is stirred frequently to insure uniform drying, and the end point is determined by noting when the sand appears to be surface-dry and free-flowing. The difference in weight of the saturated and surface-dry sand in this condition and the oven-dry weight is termed the absorption and is expressed as a percentage of the oven-dry weight. This method has been considered capable of furnishing consistent results when used by a single operator, but considerable doubt has been expressed as to the ability of different operators to check one another since the end point depends entirely on the operator's judgment. The development of a method with the end point based on measurable features was considered advisable.

In 1917, Rea presented a method of determining the so-called "apparent" specific gravity of non-homogeneous fine aggregates before the Society.[2] This value for specific gravity, more properly called the "bulk" specific gravity, is defined as "the ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature."[3] An accurate determination of this value requires that the permeable voids in the material under test be filled with water or some other substance, or that the voids be sealed against the entrance of water by some method before the volume of water displaced is determined. Rea accomplished this by coating the sand grains with kerosine before determining the volume of the test sample.

It is apparent that if the bulk specific gravity can be determined by this kerosine method, the absorption of the material tested can also be determined. By means of a slight change, the method was later adapted to determine the absorption of fine aggregate, and is described in Appendix I to the 1920 report of the Society's Committee C-9 on Concrete and Concrete Aggregates.[4] In this method, duplicate 500-g. samples of oven-dried sand are placed in 500-ml. volumetric flasks. A small amount of kerosine is added to one flask and the flask shaken to distribute the kerosine over all the sand grains. It is intended that the kerosine shall coat each sand grain to prevent absorption of water. The volume of a normal salt solution[5] required to fill the flask is then found. The excess kerosine floats on the top of the salt solution. A measured quantity of water is added to the second flask and the sand permitted to absorb water for a definite length of time. The volume of water required to fill the flask is then determined. The absorption is computed from the difference between the volume of water required to fill the flask containing the untreated sand and the volume of salt water required to fill the flask containing the kerosine-coated sand.

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[2] A. S. Rea, "Apparent Specific Gravity of Non-Homogeneous Fine Aggregates, *Proceedings*, Am. Soc. Testing Mats., Vol. XVII, Part II, p. 257 (1917).

[3] Standard Definition of Terms Relating to Specific Gravity (E 12 – 27), 1933 Book of A.S.T.M. Standards, Part I, p. 969; Part II, p. 1252.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 301 (1920).

Although this method appears to be satisfactory in theory, a number of difficulties developed in actual practice. The most troublesome feature is found when sand containing clay or other fine particles is tested. Much of this fine material adheres to the globules of surplus kerosine and floats on the salt water. Shaking the flask or stirring the liquid seems to promote the formation of an emulsified liquid containing kerosine, salt water, clay or other fine particles, and air. A clean line of demarcation between the salt water and kerosine is seldom found, and in many cases only an approximate measurement of the amount of salt water required to fill the flask can be made. For accurate determinations the test can be made only on thoroughly clean sand containing little if any fine sizes. Reports from a number of different laboratories occasionally have shown negative values for tests made by this method. The author is of the opinion that rolling and shaking the flask to remove surplus kerosine and entrapped air also tends to remove the kerosine film from the sand grains. The sand then absorbs water, and this absorption, together with the flotation of fine particles by the kerosine emulsion, may cause the volume of salt water to exceed that of the fresh water, and a negative result to be obtained.

In 1929, Pearson[6] published an article suggesting a new method of determining the absorption by sand. In this method a 200-g. sample of dry sand is placed in an Erlenmeyer flask and water added drop by drop until the finer grains adhere to the sides of the flask after thorough shaking. The weight of the flask and contents is then found, the sand dampened by several drops of water, and the sample dried back until the sand grains no longer adhere to the flask. The weight of flask and contents is again determined, and the average weight of the moistened sand is used in computing the absorption. This method has one major defect in that sufficient time for the complete absorption of the added water is not permitted. Consequently, the method may furnish results lower than the true absorption of the material under test.

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[5] The higher density of the normal salt solution gives a better separation of the kerosine than would be obtained with the use of water.

[6] J. C. Pearson, "A Simple Titration Method for Determining the Absorption of Fine Aggregates," *Rock Products*, Vol. 32, No. 10, May 11, 1929, p. 64.

In 1933, the Society adopted as tentative a method[7] for the absorption by sand, which was sponsored by Committee C-9. This method is based on Pearson's titration method. In the test, a sample of sand is thoroughly saturated with water, and dried back to a surface-dry condition as determined by visual examination. A 500-g. sample is then taken from the saturated and surface-dry sand, and placed in a 1-qt. glass jar. Water is added to the sample, drop by drop, with thorough shaking until the sand grains just tend to adhere to the sides of the jar. The weight of the sample is then found, and the sample dried to constant weight. The results of this method are in effect largely dependent on the accuracy of the operator's judgment that the sample is in a saturated and surface-dry condition. If drying the sample is stopped exactly when the sand becomes surface-dry, the addition of one or two drops of water will indicate the end point; if the sample is dried past the surface-dry condition, the addition of one or two drops of water may indicate a false end point unless sufficient time is permitted for the absorption of this water.

Chapman[8] suggested that the saturated and surface-dry condition of sand can be determined from the change in color of the material when uniformly dried, and Graf and Johnson[9] believe that this condition can be determined by drying sand for several days over calcium chloride. It is doubted that the exact point of change in color can be precisely and repeatedly determined, or that desiccation will not remove some of the absorbed water.

In addition to some of the above methods, Myers[10] presented a review and discussion of a number of methods based on gravimetric, displacement, dilution, colorimetric, and electrical-resistance principles. Many of these methods are primarily concerned with the determination of the free moisture in the aggregate, the accuracy of which depends upon placing the sand initially in a saturated and surface-dry condition by visual inspection.

Since none of the methods available was considered to combine the desirable characteristics of simplicity, ease of operation, and accuracy, an effort was made to devise a better method. Attention was first given to the possibility of mixing the sand with a material of known density and determining the density of the combined substance. A number of oils and plastic materials were tried, but the method proved unsuccessful when sands containing finely divided particles were used. Recourse was then made to the method based on Rea's kerosine test for specific gravity, and tests were conducted to find a substitute for kerosine which would furnish better results. None of the materials used gave any better results than kerosine, and further work along this line was abandoned.

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[7] Tentative Method of Test for Absorption by Aggregates for Concrete (Laboratory Determinations) (C 95-33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 711 (1933); also 1935 Book of A.S.T.M. Tentative Standards, p. 585.

[8] Cloyd M. Chapman, Discussion of Comparison of Methods of Determining Moisture in Sands, *Proceedings*, Am. Concrete Inst., Vol. XXV, p. 261 (1929).

[9] S. H. Graf and R. H. Johnson, "Study of Methods for Determining Moisture in Sand," *Prcoeedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 578 (1930).

[10] Bert Myers, "Free Moisture and Absorption of Aggregates," Report on Significance of Tests of Concrete and Concrete Aggregates, Am. Soc. Testing Mats, p. 87 (1935). Available as separate publication.

An attempt was then made to use a very simple procedure. It was known that moist sand (containing free water) could be formed into shapes by light pressure and that dry sand could not. This characteristic has long been utilized in foundries in making molds for the casting of metal objects. If this feature could be used in a method of test it might prove a satisfactory means of determining the saturated and surface-dry condition of sand. Preliminary tests were made using a small metal cone which was available. The sand was placed loosely in the cone and compacted by tamping it lightly with a metal rod having a flat face 1 in. in diameter. It was found that if the sand was dry the material would slump when the cone was was removed, but if the sand was moist the cone of sand would retain its form.

Further tests were made using right truncated cones having base angles of approximately 45, 60, 70, and 80 deg. The 45 and 60-deg. cones did not prove successful. The angles were not sufficiently different from the natural angle of repose of the sand, and a definite slump was seldom obtained, especially when the sand contained clay. The 80-deg. cone gave a sharp point of slump, but a microscopic examination of the sand disclosed the presence of an appreciable amount of free water. The 70-deg. cone also gave a definite point of slump. Microscopic examination of the sand at this point failed to show any evidence of free moisture and it is believed that the use of the 70-deg. cone determines a condition of the sand which is very close to that of the desired "saturated and surface-dry" material. The addition of a few drops of water to the test sample in the above condition caused the sand to retain its form after removal of the 70-deg. cone, thus indicating the presence of free moisture.

There seems to be considerable divergence of opinion as to the reason for the cohesion developed by the moist sand. Nutting[11] states that surface tension, vapor tension, atmospheric pressure, and internal (adsorption) pressure are the forces involved in causing the observed cohesion and resistance to deformation of moist sand. This cohesional force increases with the amount of water present up to a maximum and then falls to zero with complete wetting. Nutting further states that silica will adsorb a film of water from 50 to 100 molecules deep and that in coarse sand this water may amount to one milligram per gram of sand. However, this film of adsorbed water can be removed from sand grains only by extended desiccation or heating at an elevated temperature. The presence of this adsorbed water cannot be held the sole cause of the cohesion in damp sand since it is present in sand dried at 100 C. as well.

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[11] P. G. Nutting, "Some Mechanical Properties of Moist Granular Solids," *Journal*, Washington Academy of Sciences, Vol. 17, No. 8, April 19, 1927, p. 185.

Other physicists do not agree with these views. It is stated by one authority that absorbed water cannot be distinguished from adsorbed water, and that whether the water and the sand are physically or chemically combined, the cohesion of moist sand is due only to the presence of water on the surface of the sand grains. Another states that adsorbed moisture is present in moist sand in very small amounts as a monomolecular film, but that this water behaves as free water and joins with any other moisture present in binding the sand grains together when the damp sand is compacted. Whether the monomolecular film of adsorbed water can by itself produce the cohesion of sand is doubted.

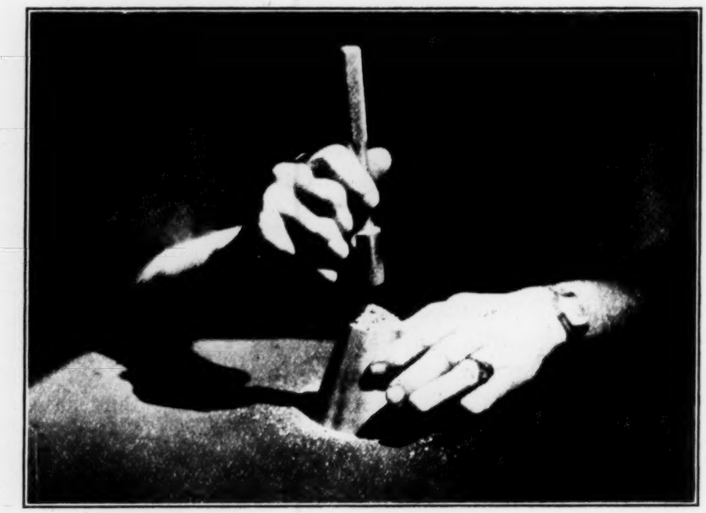
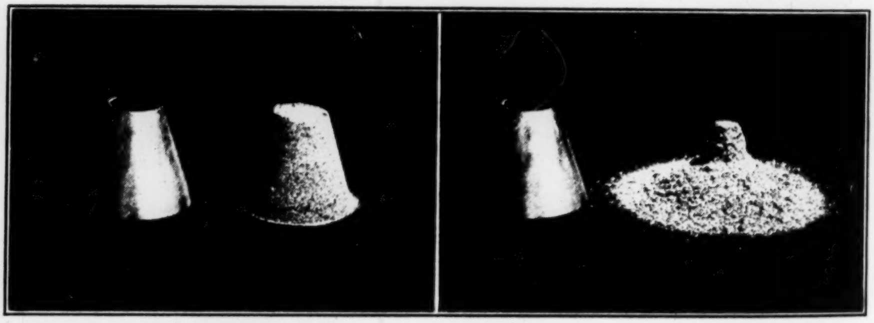


FIG. 1.—Method of Tamping Sand in the Test Cone.



(a) Sand containing free moisture. (b) Surface-dry sand.

FIG. 2.—Results of Test on Sand.

In general, the opinion seems to be that whatever the nature of the water in damp sand, a change in the cohesiveness of sand may be taken as an indication of a change in the condition of the sand from one in which free moisture exists on the surfaces of the grains to one in which no free moisture is present. Free moisture is here considered as that which causes the bulking of sand. Whether or not adsorbed moisture is present in the noncohesive sand, it seems to be generally agreed that the amount of this adsorbed water is so small as compared to the total "absorption" determined by this test that it can have no practical effect on either the bulk specific gravity or the net water-cement ratio. The test is considered to be a logical, even though possibly an empirical, method of determining a usable value by means of a readily standardized procedure.

On the basis of the preliminary tests, a method for determining the absorption by sand was prepared as follows:

> A 1000-g. sample of sand shall be placed in a pan and covered with water for a period of at least 3 hr. The sample shall then be drained, placed in a large enamelware pan, and exposed to a gentle current of warm air. The sand shall be stirred frequently to insure uniform drying. When the sand appears to be approaching a surface-dry condition, trial determinations with the cone shall be started. A sheet metal cone with top and bottom diameters of 1½ and 3½ in., respectively, and a height of 2⅞ in., shall be placed in the pan and filled with sand which shall then be lightly tamped 25 times with a 12-oz. metal rod having a flat face 1 in. in diameter. The cone shall then be lifted vertically. If the sand does not slump, free moisture is still present, and the drying shall be resumed. Trials with the cone shall be continued at frequent intervals until the sand slumps upon removal of the cone. This indicates that the sand has reached a surface-dry condition and the weight of the sample shall be determined. To insure that the sand has not been dried too much, five drops of water shall be mixed with the sand, and the cone test repeated. Under these conditions, free water should be present, and the cone of sand should not slump. The weight of the sample shall again be determined, and the sample oven-dried to constant weight. The percentage of absorption shall be computed from the average of the two weights of the dampened sand and the oven-dry weight.

Figure 1 shows the method of tamping the sand in the cone. Figure 2 (*a*) shows the cone of a sand which contains free moisture, while Fig. 2 (*b*) shows the slumped cone of the same sand after the material had reached a surface-dry condition.

In making this test, the following features should be observed:

1. The sample should be stirred frequently to obtain uniform drying.
2. The first trial for slump should find the sample with free moisture present.
3. Successive trials should be made at frequent intervals. The practice in the laboratory of the Bureau of Public Roads is to dry the sample under artificial circulation of air. As the sand approaches the surface-dry condition, drying is continued for definite lengths of time and trial determinations made at more and more frequent intervals. Under closely controlled conditions, ½-min. drying periods are used immediately prior to determination of the point of slump.

4. The metal cone should be lifted vertically. If it is not carefully removed, the sand cone may be struck and caused to slump prematurely.

One criticism offered of the cone method is that the test result obtained is influenced by the surface area of the sand grains. To investigate this, a series of tests was made with the cone method on several different gradings of five different sands. In preparing each set of test samples, a large amount of sand was separated into different sizes by sieving, and a series of samples definitely graded from coarse to fine obtained. If the cone method results were influenced by the surface area of the material, it would be expected that the test value would be increased with change in the grading from coarse to fine. As shown in Table I, however, the percentage of absorption decreased slightly with increase in fineness for each sand. These results indicate that the larger grains have a somewhat higher ratio of volume of permeable voids to volume of grain than is found in finer sand. The greater absorption of the larger grains explains the usual results of the accelerated soundness test for sand in which the coarser material usually shows the greater loss.

TABLE I.—COMPARISON BETWEEN GRADING OF TEST SAMPLE AND PERCENTAGE OF ABSORPTION.

Item	Grading of Sample, total per cent retained									
Sieve No. 4	2	0	0	1	0	1	0	0	0	0
Sieve No. 8	24	25	14	12	8	8	0	0	0	0
Sieve No. 16	46	47	30	27	17	17	4	4	0	0
Sieve No. 30	80	80	73	70	60	60	45	45	40	40
Sieve No. 50	95	95	93	90	85	85	75	75	70	70
Sieve No. 100	100	100	100	97	100	95	100	93	100	90
Fineness modulus	3.47	3.47	3.10	2.97	2.70	2.66	2.24	2.17	2.10	2.00
	Absorption, per cent									
River sand No. 1	1.19	......	......	1.19	......	1.17	......	1.15	......	1.14
River sand No. 2[a]	......	2.1	1.8	......	1.8	......	1.7	......	1.6	......
Pit sand No. 1	......	2.1	......	......	1.9	......	......	......	1.7	......
Pit sand No. 2	......	1.9	......	......	1.8	......	......	......	1.5	......
Pit sand No. 3	......	0.4	......	......	0.4	......	......	......	0.3	......

[a] Each value is the average of two tests.

TABLE II.—SANDS USED IN COOPERATIVE TESTS.

Sand	Source	Mineralogical Composition
No. 1	Massachusetts	Quartz, granite, diorite, rhyolite, feldspar sandstone
No. 2	New York	Limestone, sandstone, quartz, chert
No. 3	South Carolina	Quartz
No. 4	Kansas	Quartz, feldspar, chert
No. 5	Rhode Island	Quartz, feldspar, granite, slate
No. 6	Ohio	Limestone (crushed stone sand)

Following the establishment of a presumably satisfactory method of test, a large number of tests was made by different operators in the laboratory. The results of these tests were considered so promising that it was decided to submit this method to a number of other laboratories for their criticisms. Several laboratories were accordingly invited to cooperate with the Bureau in an investigation of methods of determining absorption. The following laboratories cooperated in the investigation:

Duquesne Slag Products Co.
Indiana State Highway Commission
Iowa State Highway Commission
Michigan State Highway Dept.
National Crushed Stone Assn.
National Sand and Gravel Assn.
Pennsylvania State Highway Dept.
Portland Cement Assn.
Rhode Island State Board of Public Roads
U. S. Bureau of Public Roads
Virginia Department of Highways
West Virginia State Road Commission

In this series of cooperative tests, each laboratory was requested to make five tests by each of four different methods on each of six different samples of fine aggregate. The methods are included in those previously described.

Method No. 1.—Kerosine method,
Method No. 2.—A.S.T.M. Tentative Method C 95 - 33 T,[7]
Method No. 3.—Cone method, and
Method No. 4.—Visual-inspection method.

The samples of fine aggregate used in these tests were obtained by the Bureau, mixed thoroughly, and suitable portions of each sent to the cooperating laboratories. Effort was made to obtain sands having a considerable range in absorption, and the samples selected were as shown in Table II.

The results of this series of cooperative tests are shown in Table III and Figs. 3 and 4. With the exception of sand No. 3, the values obtained by the use of the kerosine method are considerably lower than those for the other test methods. This may be caused by the removal of a portion of the kerosine from the sand grains when the flask is shaken to free the excess kerosine, or it may be due to retention of a portion of the sample by the floating kerosine. In either case the result is the same in that an excessive quantity of salt water would be added to the flask. This would of course decrease the difference between the volumes of fresh and salt water and furnish a low value for the percentage of absorption. The average values with the cone method are found to be slightly higher than those with the A.S.T.M. tentative method or visual-inspection methods, but the difference in results is marked only in the case of the stone sand sample No. 6.

It is possible that the extreme angularity of the stone sand was the determining factor in causing the higher value in the cone test than in the A.S.T.M. or visual-inspection tests. It is also possible that there may have been some cementing of the particles by the dust present in the material. On the other hand, the dust present in the stone sand may have had such an affinity for moisture that the A.S.T.M. and visual-inspection tests continued to show water present although the larger portion of the sample was only partially saturated. This would account for the lower values obtained by the use of these two methods.

TABLE III.—COOPERATIVE ABSORPTION TESTS ON SAND.

Method	Average Values for Percentage of Absorption												Average
	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3	Laboratory No. 4	Laboratory No. 5	Laboratory No. 6	Laboratory No. 7	Laboratory No. 8	Laboratory No. 9	Laboratory No. 10	Laboratory No. 11	Laboratory No. 12	
SAND NO. 1													
No. 1	0.2	0.60	0.93	0.6	0.6	0.26	0.80	0.33	0.67	0.41	0.34	0.18	0.49
No. 2	0.7	0.83	0.94	0.6	0.4	0.73	0.69	0.93	0.50	0.24	0.79	0.43	0.65
No. 3	0.8	1.11	0.76	0.8	0.8	0.89	0.77	0.88	0.78	0.55	0.77	0.74	0.80
No. 4	0.7	1.00	0.76	0.6	0.9	0.66	0.56	0.40	0.72	0.42	0.77	0.37	0.65
SAND NO. 2													
No. 1	0.5	1.06	0.53	0.6	0.9	0.51	......	0.70	1.06	0.92	0.66	0.58	0.73
No. 2	1.7	1.06	1.62	1.4	0.5	2.08	1.84	1.53	1.13	1.30	1.62	1.20	1.42
No. 3	1.8	2.15	1.56	1.6	1.2	1.59	1.60	1.65	1.57	1.19	1.58	1.41	1.57
No. 4	1.7	1.59	1.55	1.4	1.2	1.24	1.48	1.37	1.38	1.31	1.61	1.05	1.41
SAND NO. 3													
No. 1	0.2	0.55	0.80	0.2	0.5	0.27	......	0.18	0.29	0.25	0.42	0.10	0.34
No. 2	0.2	0.67	0.25	0.3	0.4	0.08	0.10	0.21	0.12	0.16	0.21	0.14	0.24
No. 3	0.2	0.70	0.25	0.2	0.5	0.20	0.22	0.24	0.21	0.13	0.24	0.18	0.27
No. 4	0.2	0.42	0.24	0.2	0.8	0.12	0.19	0.13	0.22	0.12	0.23	0.12	0.25
SAND NO. 4													
No. 1	0.1	0.40	0.76	0.0	0.2	0.07	......	0.13	0.41	0.52	0.36	0.14	0.28
No. 2	0.6	0.69	0.71	0.6	0.4	0.65	0.58	0.47	0.43	0.57	0.61	0.53	0.57
No. 3	0.7	0.62	0.56	0.6	0.7	0.51	0.73	0.71	0.61	0.56	0.65	0.55	0.62
No. 4	0.7	0.84	0.53	0.6	0.6	0.52	0.53	0.39	0.66	0.49	0.63	0.38	0.57
SAND NO. 5													
No. 1	0.1	0.31	1.10	0.2	0.6	0.38	......	0.23	0.42	0.62	0.44	0.10	0.41
No. 2	0.7	0.79	0.69	0.6	0.5	0.86	0.54	0.54	0.40	0.48	0.73	0.45	0.61
No. 3	0.7	0.30	0.69	0.7	0.9	0.64	0.62	0.78	0.70	0.57	0.79	0.77	0.68
No. 4	0.7	1.54	0.58	0.6	0.9	0.48	0.63	0.54	0.73	0.38	0.72	0.38	0.68
SAND NO. 6													
No. 1	0.5	1.48	0.39	0.7	0.7	0.47	......	1.85	1.10	1.01	0.79	0.62	0.87
No. 2	1.5	1.37	1.45	1.1	0.3	0.88	1.32	1.33	1.02	1.40	1.67	0.82	1.18
No. 3	1.7	2.11	1.66	1.7	1.3	1.31	1.82	1.98	1.63	1.16	1.42	1.14	1.58
No. 4	1.4	1.47	1.39	1.2	1.0	1.44	1.56	1.57	1.09	1.16	1.48	0.55	1.28

In Fig. 3, the percentage deviation of the average result for each laboratory from the average for all laboratories is given for each method. Each value is an average for all six sands, except that the reported value for laboratory No. 7 for the kerosine method (No. 1) is for one sand only. These values show that the cone method (No. 3) furnishes test results which are most nearly duplicated by different laboratories. Using the cone method, only one laboratory (No. 2) shows a large deviation from the average, and

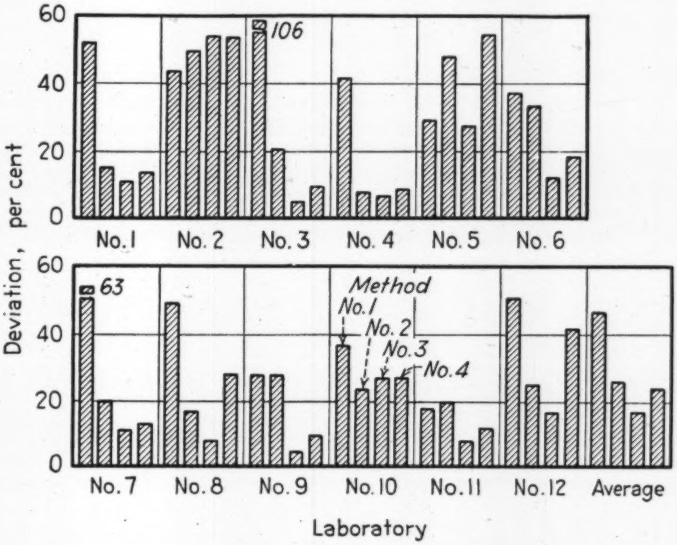


FIG. 3.—Deviation of Average Result for Each Laboratory from Average for All Laboratories. Average values for all sands.

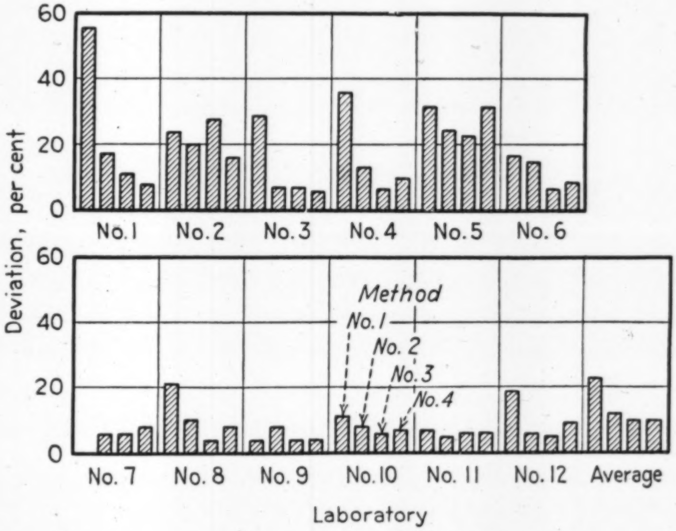


FIG. 4.—Average Deviation of Individual Results Obtained in Each Laboratory from Average Results for That Laboratory.

eight of the twelve laboratories have deviations of 15 per cent or less. The average for all laboratories is 17 per cent. The visual-inspection method appears to be the second most accurate. Three laboratories show wide variations from the average but six have variations of 15 per cent or less. The average deviation is 24 per cent. The A.S.T.M. tentative method (No. 2) gives about the same average variation (26 per cent) as the visual-inspection method, but only two laboratories have deviations of 15 per cent or less, while seven laboratories show deviations of 16 to 30 per cent. The kerosine method is the least accurate of those under consideration. Only two laboratories show deviations of less than 30 per cent and results deviating from the average by over 50 per cent were found in four laboratories. The average value is 47 per cent.

The deviations within a given laboratory as shown in Fig. 4 indicate that concordant results are usually obtained by the use of the A.S.T.M., cone, and visual-inspection methods. In most cases the kerosine method fails to furnish results agreeing with one another to a satisfactory degree.

In considering the entire series of tests, the cone method appears to be the most satisfactory means of determining the absorption by sand. It furnishes more concordant results between different laboratories than any of the other methods under consideration, and permits an operator to check his own work with an average variation of less than 10 per cent. The average values for percentage of absorption found by this method agree very closely with those of the A.S.T.M. and visual-inspection methods. The A.S.T.M. and visual-inspection methods give very good agreement between the results of tests made by a single operator and fair agreement between the results of different laboratories. The kerosine method is found to give low and very erratic results and the further use of this method is not recommended.

The cone method has been adopted by the American Association of State Highway Officials for use in its standard method of test for specific gravity and absorption of fine aggregate.[12] It has also been adopted tentatively by the Joint Committee on Methods of Test for Specific Gravity of Aggregates of the A.S.T.M. Committees C-9 on Concrete and Concrete Aggregates and D-4 on Road and Paving Materials for use in connection with the committees' recommendations regarding the unification of the various Society methods for determining specific gravity. These recommendations are now before the two committees for consideration.

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[12] Method T-84, Am. Assn. State Highway Officials Book of Standard Specifications for Highway Materials and Methods of Sampling and Testing (1935).

DISCUSSION

MR. IRA PAUL[1] *(presented in written form)*.—In a paper on the "Magnesium Sulphate Accelerated Soundness Test on Concrete Aggregates," which I presented before the Highway Research Board in December, 1932,[2] I described a new laboratory method for determining the absorption of sands. This new method is based on the Rea kerosine method minus the undesirable features described by Woolf.

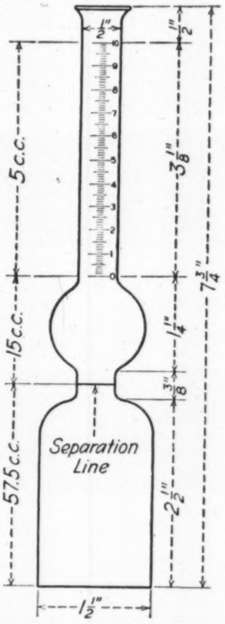

FIG. 1.—Absorption Bottle for Sands.

Special apparatus consisting of a specially designed combination of a Le Chatelier flask and Babcock bottle was developed for this purpose (Fig. 1). The bottle was designed and calibrated so that direct readings on a percentage basis can be obtained. The lower portion of the bottle has a volume of 57.5 ml. The upper part has a bulge holding 15 ml. and a tube with a volume of 5 ml. The scale on the tube is divided into 100 parts so that each division is equivalent to 0.05 ml. An electric centrifuge capable of developing a speed of 600 r.p.m. completes the equipment necessary to make a test.

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[1] Associate Materials Engineer, New York State Department of Public Works, Albany, N. Y.

[2] Ira Paul, "Magnesium Sulfate Accelerated Soundness Test on Concrete Aggregates," *Proceedings*, Highway Research Board, Vol. 12, p. 322 (1932).

The method for determining the absorption on sands is as follows:

> Wash thoroughly a representative sample of about 300 g. of the fine aggregate on a No. 100 sieve. Thoroughly dry the sample on the sieve at 105 C. Weigh 50 g. of the washed and dried sand into a dry absorption bottle, and add 20 ml. of water white kerosine from a burette. Let stand for 24 hr. Then add a saturated solution of sodium chloride, filling the bottle to the middle of the bulb. Tilt the bottle as nearly horizontal as possible, not allowing the liquid to flow up into the graduate tube. Rotate the bottle slowly until sand on the bottom of the bottle flows freely. Centrifuge for five minutes at 560 r.p.m. Remove the bottle from the centrifuge and rotate as before. Again centrifuge for five more minutes at 560 r.p.m. Bring the meniscus between the salt solution and kerosine to the separation line on the bottle, by the addition of more salt solution. Read upper meniscus on tube directly for the percentage of absorption by the sand.

A comparative series of tests was made in our laboratory between the cone method and the modified kerosine method, with the results shown in the accompanying Table I. Eighteen samples of sand ranging from our best type to the poorest quality were used in this test series.

Judging from the results obtained, the two methods compared favorably. An analysis of the results showed eleven samples with slightly higher absorption results with the modified kerosine method. Four samples were higher with the cone method, and three samples gave the same results with both methods. The removal of the fines passing the No. 100 sieve by washing had practically no effect in the modified kerosine method. In the eighteen samples tested the fines passing the No. 100 sieve ranged from 1.7 to 5.9 per cent.

A check on three samples of sand from the same source of supply taken at different intervals gave exactly the same percentage of absorption of 1.1 per cent with the modified kerosine method; whereas, the cone method varied from 0.9 to 1.3 per cent. A sample of dredged lake sand of good quality showed 1.9 per cent with the cone method and 1.5 per cent with the modified kerosine method. A poor quality sand taken from a pit had 3.2 per cent absorption by the cone method and 2.6 per cent with the modified kerosine method. With some sands we found that this variation was due to the fact that other factors entered into the cone method for the determination of the slump point. Some sands slump quicker than others depending upon the characteristics and shape of the sand grains. Secondly, the cone method is dependent upon the surface tension of the moisture to hold the sand particles together. No personal factors enter into the modified kerosine method of test. Readings are made in the same way as the dairy chemist makes a Babcock test for fat content in milk or cream.

With the modified kerosine method about 30 min. is involved in making the absorption test on four samples; whereas, the cone method requires a half day's time of the operator to obtain results on three samples.

We have been using the modified kerosine method for determining the percentage of absorption by sands for over four years and have made several hundred of tests. The undesirable features referred to by Woolf, as well as personal factors in a test of this type, have been eliminated from the original kerosine method in the manipulation as described.

TABLE I.—RESULTS OF ABSORPTION TESTS ON SAND.

Sample	Passing No. 100 Sieve, per cent	Absorption, per cent	
		Cone Method	N.Y.S. Modified Kerosine Method
No. 1	2.3	0.42	0.5
No. 2	1.9	0.6	0.7
No. 3	4.2	0.7	0.7
No. 4	1.9	0.5	0.8
No. 5	3.0	0.9	1.1
No. 6	3.2	1.3	1.1
No. 7	2.4	1.1	1.1
No. 8	3.4	0.8	1.1
No. 9	5.9	0.82	1.0
No. 10	5.2	1.1	1.2
No. 11	5.5	1.05	1.2
No. 12	1.7	1.9	1.5 check 1.5
No. 13	3.0	1.8	1.8
No. 14	4.6	1.75	1.8
No. 15	2.6	1.9	2.0
No. 16	5.1	2.4	2.35
No. 17	3.1	3.2	2.6 check 2.5
No. 18	3.7	2.8	3.0

MR. W. H. HERSCHEL.[3]—I should like to ask Mr. Woolf if his cone method will work with a small amount of clay in the sand? In his paper he very distinctly brought out that most methods that have previously been used have failed when there is fine material present.

MR. D. O. WOOLF.[4]—The amount of clay and other fine particles normally present in concrete sand causes no difficulty in the use of either the cone method or the visual inspection method. Some difficulty may be found if sand containing fine particles is tested by the A.S.T.M. bottle method, and sand tested by the kerosine method should be washed free of clay.

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[3] Associate Physicist, National Bureau of Standards, Washington, D. C.

[4] Associate Materials Engineer, Division of Tests, U. S. Bureau of Public Roads, Washington, D. C.

INTERNAL STABILITY OF GRANULAR MATERIALS

By William S. Housel[1]

Synopsis

The paper discusses inconsistencies which have arisen in internal friction theories and presents the results of a series of tests on five different granular materials. In these data there is no direct relation between shearing resistance, or friction, and the principal pressures which express internal stability in a granular mass. Shearing resistance is measured by direct shear tests and the principal pressures are measured by a specially designed stabilometer test briefly described in the paper.

A general equation for ultimate bearing capacity is derived as well as an equation to represent pressure distribution and capacity at loads less than ultimate. The results of a comprehensive series of bearing-capacity tests on the same materials used in other types of tests are presented and correlated with the internal-stability relations.

Internal stability is defined as that mechanical property of granular masses which produces resistance to displacement by the mutual support of adjacent particles in the mass, involving static forces and reactions between particles too large to be noticeably affected by molecular forces.

The paper concludes that the behavior of granular materials under stress can be accurately and simply described in terms of the stability of elementary arches of soil particles in which the ability to sustain vertical pressure is dependent on the horizontal thrusts supplied by adjacent particles in the mass. The relation between the vertical and lateral principal pressures depends simply upon the angle of transmission between the arch axes or lines of contact between particles and the vertical axis of reference. Such a system is statically determinate to a degree not possible when the internal resistance is pictured as resistance to sliding on some inclined plane. The "arch action" conception is applied to three basic problems: first, the distribution of load concentrations through an indefinite granular mass; second, the relation between the principal pressures in a granular mass; and third, the bearing capacity of a granular mass including a satisfactory description of the manner in which failures occur under load concentration.

The theory of the stability of earth masses and of granular materials in particular which is recounted herein had its origin in observations and measurements of earth pressure and stability of deep foundations carried out in the City of Detroit by the University of Michigan and others.[2] In the course of this investigation the analysis of stress components in a mass of earth as observed with load tests on steel cylinder piles led to the formulation of a tentative hypothesis, relating the principal pressures in an earth mass.[3] This formulation may be termed an "arch theory" of internal stability and appears to be a consistent and logical method of describing the mechanics of internal resistance of granular masses. During the past three years additional investigations have been made in the State Highway Laboratory with the assistance of the Michigan State Highway Department. Apparatus has been developed and tests conducted by Donald S. Berry, Detroit Edison Fellow in Highway Engineering, University of Michigan, the preliminary report of which was submitted to the Society in 1935.[4]

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[1] Research Consultant, Michigan State Highway Dept.; Associate Professor of Civil Engineering, University of Michigan, Ann Arbor, Mich.

[2] Other organizations cooperating in the investigation of soil properties include the Detroit Edison Co.; Wayne County Road Commission; City of Detroit, Department of Buildings and Safety Engineering, Department of Public Works, Department of Water Supply, and Rapid Transit Commission.

INTERNAL-FRICTION THEORIES

Friction:

In proposing a group of conceptions to take the place of existing theories of stability based primarily on internal friction, it is first necessary to consider certain fundamental inconsistencies which have become increasingly apparent in those theories. In the first place, friction is characteristic of a state of motion and is not basically sound when used to describe conditions of equilibrium or states of rest. Upon first thought it might appear that such a statement ignores the commonly accepted static friction. However, Rankine[5] in the original development of the Rankine theory when discussing the excess of static friction over the friction of motion said: "That excess, however, of the *friction of rest* over the *friction of motion* is instantly destroyed by a slight vibration; so that the *friction of motion* is alone to be taken into account as contributing to the stability of a structure." It seems apparent that, when the structure considered is an arrangement of irregularly shaped particles interlocked across an assumed plane of sliding, there must be a static resistance of a quite different character than the friction of motion which serves as the basis of internal friction theories. It also seems apparent that this static resistance in the interior of a compact granular mass cannot be destroyed by mere vibration. In fact, as pointed out by Terzaghi,[6] it cannot be overcome without introducing a stage of volume expansion which would permit the particles to roll over one another. Experiment has shown that this tangential resistance is not always directly proportional to normal stress components.

Planes of Failure:

A second inconsistency in internal-friction theories may be pointed out in the contradiction between the basic assumptions on which the theories are founded and the resulting conclusions as to the manner in which failure takes place. In describing the conditions of stress in a granular mass in the same manner as is used in homogeneous elastic solids it is assumed that the material is isotropic. In this case the condition is one of statistical isotropy, as described by Krynine,[7] but nevertheless the condition assumed presupposes the same ability to resist displacement in all directions. After the stress conditions under which failure takes place have been deduced from the application of the laws of equilibrium to determine the difference between the greatest and least principal pressure, it is found that the plane of failure is not the plane of maximum shearing stress. Even though the theory assumes that the failure takes place in shear the failure plane is one of minimum resistance and does not coincide with the plane of maximum stress. This situation is equivalent to the introduction of preferential planes of weakness and violates the assumption of isotropy.

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[3] W. S. Housel, "Bearing Capacity of Steel Cylinder Piles," Unpublished report submitted to Detroit Edison Co., July, 1933.

[4] D. S. Berry, "Stability of Granular Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 491 (1935).

[5] W. J. M. Rankine, "Manual of Civil Engineering," p. 171, Griffin, Bohn and Co., London (1862).

[6] K. Terzaghi, "Old Earth Pressure Theories and New Test Results," *Engineering News-Record*, Vol. 85, p. 637 (1920).

Angle of Repose:

The third objection which might be raised to existing practice in the application of internal friction to stability arises from the confusion of the angle of repose and the angle of internal friction. Coulomb in the development of the "wedge" theory, which differs from the Rankine theory only in the field of application, specifically stated that the angle of repose and the angle of internal friction were entirely different. Nevertheless, Rankine assumed that they were the same and it is common practice at the present time to use the angle of repose as a measure of the angle of internal friction. It is comparatively simple, as will be shown later, to demonstrate the error in this mistaken conception and several investigators since Coulomb have pointed it out.[8]

INTERNAL STABILITY

There are several other aspects of the problem of internal friction which are not satisfactorily treated by orthodox theories, but it does not appear necessary to undertake a more exhaustive analysis in this paper. However, in the light of the inconsistencies that have been pointed out it does appear quite desirable to restate the mechanics of internal stability involving, perhaps, some different, if not entirely new, conceptions. The "arch theory" is proposed in an attempt to so restate the problem and to define stability in such a way as to eliminate the objections in the existing theories. If such an undertaking is to be successful it is necessary that the conceptions of internal resistance introduced shall form an adequate basis for describing three basic phenomena which constitute problems of considerable importance. These are: first, the distribution of load concentrations through an indefinite granular mass; second, the relation between the principal pressures in a granular mass; and third, the bearing capacity of a granular mass including a satisfactory description of the manner in which failures occur under load concentration.

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[7] Discussion by D. P. Krynine, "Distribution of Stresses Under a Foundation," *Proceedings*, Am. Soc. Civil Engrs., Vol. 61, October, 1935, p. 1253.

[8] J. Feld, "Determination of Lateral Earth Pressure," *Transactions*, Am. Soc. Civil Engrs., Vol. 86, p. 1451 (1923).

Internal stability is defined as that mechanical property of granular masses which produces resistance to displacement by the mutual support of adjacent particles in the mass, involving static forces and reactions between particles too large to be noticeably affected by molecular forces. Internal stability is contrasted with cohesion which is another basic property necessary to a complete description of the mechanics of soil resistance in general.

Cohesion is defined as that property of a material which produces resistance to displacement by the mutual attraction between particles involving forces of molecular origin, characteristic of microscopic and submicroscopic matter. This definition is intended to include both cohesion and adhesion as ordinarily defined.

The behavior of granular materials under stress can be accurately and simply described in terms of the stability of elementary arches of soil particles in which the ability to sustain vertical pressure is dependent on the horizontal thrusts supplied by adjacent particles in the mass. The relation between the vertical and lateral principal pressures depends simply upon the angle of transmission between the arch axes or lines of contact between particles and the vertical axis of reference. Such a system is statically determinate to a degree not possible when the internal resistance is pictured as resistance to sliding on some inclined plane. The "arch action" conception can be applied directly to the three basic problems outlined and this application will be the basis of the subsequent discussion. The author has chosen to treat these problems upon a theoretical basis using typical test results for illustration as well as to produce experimental evidence supporting the theory.

DISTRIBUTION OF CONCENTRATED LOADS

The lateral distribution of vertical pressure beneath load concentrations on the surface of an indefinite mass has been investigated so thoroughly[9] during the development of modern soil mechanics that it is possible to limit this discussion to those features of pressure distribution which are related to arching action in granular masses. It is generally recognized that the normal pressure distribution curves may be determined by the law of probabilities applied to the unlimited number of probable paths which pressure may follow in passing through an aggregation of soil particles. Various conditions and systems of mechanical arrangement have been used to picture the manner in which this distribution takes place. One of the most common of these is to represent the grains in the mass as spheres of uniform size corresponding to an average shape and size of the

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[9] A. E. Cummings, "Distribution of Stresses Under a Foundation," *Proceedings*, Am. Soc. Civil Engrs., Vol. 61, August, 1935, p. 823. Discussion by C. C. Williams, D. P. Krynine, and L. C. Wilcoxen, October, 1935, p. 1251; M. G. Findley, and M. A. Biot, November, 1935, p. 1398; J. Feld, G. Paaswell, and G. S. Salter, December, 1935, p. 1546; W. S. Housel, and N. M. Newmark, Vol. 62, January, 1936, p. 142.

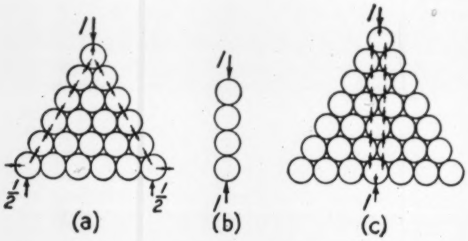
1
1/2
1/2
(a)
1
1
(b)
1
1
(c)

FIG. 1.—Pressure Distribution by Unstable Equilibrium.

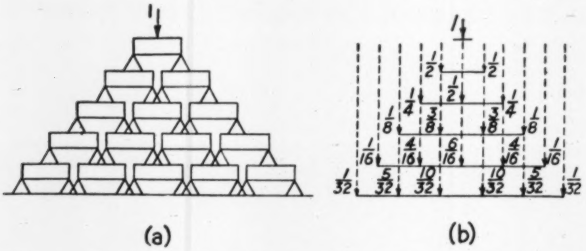
1
(a)
1
1/2
1/2
1/2
1/4
3/8
3/8
1/4
1/8
4/16
6/16
4/16
1/16
1/32
5/32
10/32
10/32
5/32
1/32
(b)

FIG. 2.—Pressure Distribution in Simply Supported Beams.

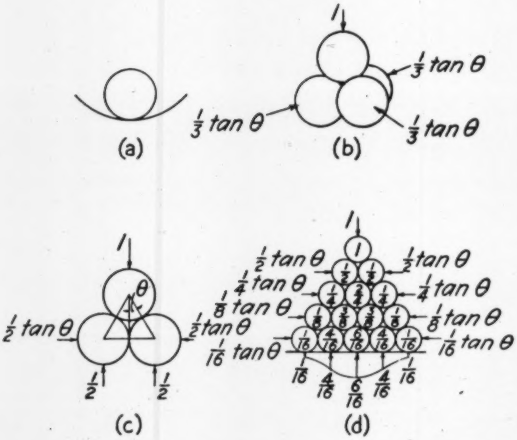
(a)
1
1/3 tan θ
1/3 tan θ
1/3 tan θ
(b)
1
θ
1/2 tan θ
1/2 tan θ
1/2
1/2
(c)
1
1/2 tan θ
1/2 tan θ
1/4 tan θ
1/4 tan θ
1/8 tan θ
1/8 tan θ
1/16 tan θ
1/16 tan θ
1/16
4/16
6/16
4/16
1/16
(d)

FIG. 3.—Pressure Distribution Under Stable Equilibrium.

more or less irregular shapes and non-uniform sizes which actually exist. In order to simplify the relationships involved in the variation in mechanical arrangement, shape, and size of particles, the author prefers this device and has used it extensively in experimental work as well as in exposition.

Unstable Equilibrium:

In Fig. 1 is illustrated the distribution of a concentrated load on a pyramid of spheres as pictured by Froehlich.[10] It is contended as shown in Fig. 1 (*a*) that lines of transmission through such an arrangement introduce radial forces which concentrate the load down two lines of spheres instead of distributing it in a normal manner.

It should be pointed out that such a concentration of force along one path rests on the uncertainties of unstable equilibrium as illustrated in Fig. 1 (*b*) and is practically precluded by a number of existing conditions. No such system could be permanent under the slightest vibration or eccentricity. Further than this there is no basis for assuming that the horizontal thrust necessary for stable equilibrium, which must be supplied by particles in the surrounding mass, will select any specific horizontal layer as more capable than any other in supplying this thrust.

Froehlich concludes, as shown in Fig. 1 (*c*), that the introduction of friction between spheres may approach the necessary condition of transmitting load to those spheres immediately beneath the concentrated load, where experimental evidence shows that the pressure ordinates are largest. It is conceded, however, that friction alone is not sufficient to produce the observed result and a pyramid of simply supported beams shown in Fig. 2 is selected as the system which will represent a normal law of pressure distribution.

Such a system of horizontal beams is entirely artificial and introduces conditions which are not natural representations of mechanical arrangements of a mass of granular particles. This is particularly evident when it is pointed out that in such a system lateral pressures would be zero while in a granular mass lateral pressure is a primary necessity for equilibrium.

Stable Equilibrium:

The introduction of the conditions of stable equilibrium in the mass immediately clarifies the situation leading to a normal distribution of vertical pressure with the lateral pressure represented in accordance with the natural conditions necessary for equilibrium. These conditions are shown in Fig. 3.

In Fig. 3 (*a*) a typical case of stable equilibrium is illustrated as a sphere resting in spherical shell. The particle is said to be in stable equilibrium because any displacement due to vibration or application of force generates a reacting force tending to equilibrium and upon removal of the activating influence the sphere returns to its normal position.

In Fig. 3 (*b*) is shown an elementary arch representative of a system of granular particles in which any specific grain rests in the depression between several other grains in the layer beneath it. The horizontal thrust necessary to stable equilibrium is supplied by adjacent particles in each layer with no preference shown for more horizontal resistance in one layer than another. Thus the vertical force transmitted to any one particle is divided equally among the supporting particles which in turn distribute their vertical components in a like manner. For the sake of simplicity the description of pressure distribution through such a mass may be represented in two dimensions as in Fig. 3 (*c*) in which the angle θ denotes the angle of pressure distribution along the arch axis through the center of the primary and supporting spheres.

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[10] Discussion by O. K. Froehlich of Progress Report on Earths and Foundations, of Committee on Bearing Value of Soils for Foundations, *Proceedings*, Am. Soc. Civil Engrs., Vol. 59, November, 1933, p. 1470.

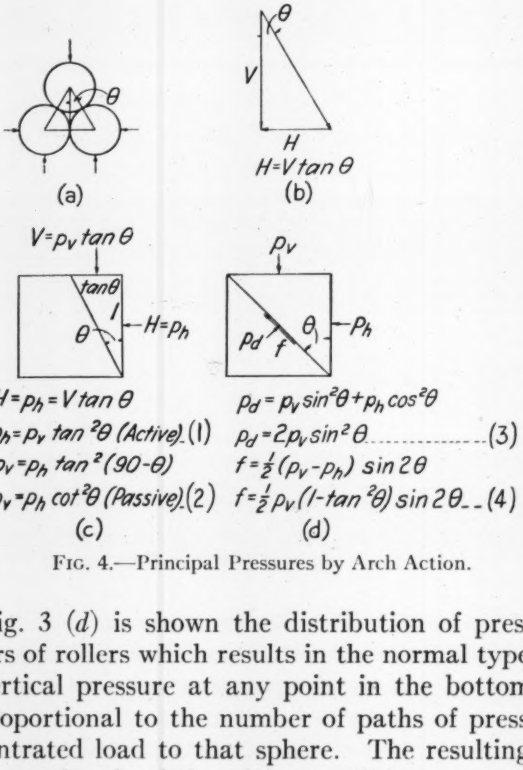


FIG. 4.—Principal Pressures by Arch Action.

Thus in Fig. 3 (*d*) is shown the distribution of pressure through a number of layers of rollers which results in the normal type of distribution curve. The vertical pressure at any point in the bottom layer may be shown to be proportional to the number of paths of pressure which lead from the concentrated load to that sphere. The resulting regular distribution curves may then be deduced from well-known statistical laws.

In the example shown the angle of distribution θ is 30 deg. and corresponds to the most compact arrangement of rollers that it is possible to obtain. In natural granular systems the elementary arch structure will vary depending upon the mechanical arrangement in three dimensions for various degrees of compaction. Such departures from the example given would result in an increase in the volume of the mass which could be represented by an increase in void space and a corresponding increase in the angle θ.

The tendency in any natural system is toward a mechanical arrangement resulting in a minimum volume. Therefore, the examples used in the present discussion have been chosen to represent the most compact arrangement as more representative of experiments, most of which have been made at the maximum compaction possible by mechanical vibration.

PRINCIPAL PRESSURES IN A GRANULAR MASS

Conditions of Stability:

The condition of equilibrium which exists in the interior of an indefinite granular mass has long been a problem of primary importance and was one of the first problems in soil mechanics to engage the attention of engineers and mathematicians. The solution of this problem in any case involves a determination of the relation between the greatest and least principal pressure, but the conditions controlling this relation have always been a matter of some controversy. In the present development the author proposes to describe the equilibrium of an element within the mass in terms of the stability of elementary arches of soil particles.

In Fig. 4 the relations essential to this development are shown. In Fig. 4 (*a*) is shown the elementary soil arch and the angle of pressure transmission θ which determines the amount of the horizontal thrust necessary to the equilibrium of any soil arch carrying vertical force. Figure 4 (*b*) is the force diagram with the horizontal thrust determined as equal to the vertical component multiplied by tan θ ($H = V \tan \theta$). In Fig. 4 (*c*) is shown an elementary cube of unit dimensions taken from the interior of the granular mass, from which the relation between pressure on a horizontal plane and pressure on a vertical plane is shown. The total vertical force V acting on that portion of the horizontal plane which is held in equilibrium by horizontal thrust on the vertical face of unit area, is p_v tan θ. By substitution, the equation for the relation of principal pressures then becomes:

$$p_h = p_v \tan^2 \theta \dots\dots\dots\dots\dots\dots\dots\dots (1)$$

It should be pointed out that the shearing force acting on the inclined plane parallel to the direction of force transmission plays no part in the relation between the intensity of horizontal thrust or lateral pressure and the vertical pressure. There is, of course, a tangential component and a normal component on the inclined plane, both of which bear a constant ratio to the principal pressures for all intensities of these pressures while equilibrium is maintained. The same statement applies to tangential components on the plane of maximum shear at 45 deg. with the principal planes. The relations shown in Fig. 4 (*d*) demonstrate that for any given value of θ the maximum shearing stress remains equal to one half the difference between the principal pressures and cannot exceed this critical value as long as structural stability of the soil arch is maintained. The stability of the soil arch, in turn, cannot be disturbed except by applying a vertical pressure which exceeds the critical value as controlled by the horizontal thrust available as support from an adjacent element, or on the other hand, applying a lateral force sufficient to overcome the stability due to the vertical pressure as the reacting pressure.

When failure does take place the first lines of motion naturally will be in the direction of force transmission parallel to the arch axes. It appears, however, that after the first movement the direction of displacement changes and any critical plane of sliding within the mass is indeterminate. When failure is due to exceeding the lateral pressure available as horizontal thrust, the lateral movement of supporting particles will result in flattening of the soil arch. The increasing values of θ would require a corresponding increase in horizontal thrust which has already been exceeded and failure would be sudden and complete unless additional resistance were supplied from some source.

It appears at the present time that there may be additional resistance developed by re-orientation of particles after some movement and adjustment has taken place. The probable sources of additional resistance will be pointed out in the discussion of experimental results and seem to be most satisfactorily described by the variations in the angle of pressure transmission which have been noted.

Active and Passive Pressure:

No description of internal stability of granular materials would be complete without a discussion of active pressure and passive pressure or passive resistance. In Fig. 4 (*c*) there is only one condition of equilibrium portrayed rather than two limiting conditions as in the conventional conception of active and passive pressure. The maximum principal pressure corresponds to what has been called passive resistance and the minimum principal pressure represents the active pressure as ordinarily defined. The relation between these pressures is constant and defined by the angle θ, or its complement (90 deg. $- \theta$). The relative stability may be described in terms of either one of the principal pressures, depending upon which is shown as the applied pressure, the other then being the reacting pressure or passive resistance. Whether the passive resistance is greater than the active pressure depends entirely upon the mechanical arrangement and whether the direction of applied pressure is such that the angle θ has a minimum value. Examples of the effect of varying the angle θ will best serve to illustrate these relations.

Case 1, $\theta = 0$ deg.—When θ is equal to 0 deg., the lateral pressure is zero and the vertical pressure is infinite as far as internal stability is concerned. In terms of arch action this case represents a cohesive material or a solid in which vertical pressure may be sustained within the limits of cohesion without the necessity of lateral support. The following equations represent this condition:

$$p_h = p_v \tan^2 \theta \qquad (1)$$

$$p_v = p_h \cot^2 \theta \qquad (2)$$

when

$$\theta = 0 \text{ deg., } \tan^2 \theta = 0, \text{ and } \cot^2 \theta = \infty$$

Therefore

$$p_h = 0 \text{ and } p_v = \infty$$

Case 2, $\theta = 30$ *deg.*—When θ is equal to 30 deg., the lateral pressure is the minimum principal pressure and has a value of one third of the vertical pressure. This value corresponds to the usual active pressure. The vertical pressure has a value of three times the lateral pressure and corresponds to what has been known as passive resistance:

when

$$\theta = 30 \text{ deg., } \tan \theta = 0.577, \text{ and } \tan^2 \theta = \tfrac{1}{3}, \cot^2 \theta = 3$$

therefore

$$p_h = \tfrac{1}{3}\, p_v \text{ and } p_v = 3\, p_h$$

Case 3, $\theta = 45$ *deg.*—A value of θ of 45 deg. represents an equality of vertical and lateral pressures analogous to fluid pressure. There are no tangential components on any inclined surface and normal components are equal to the principal pressures. As discussed later, this case represents internal stability in a dispersed clay in which the voids are completely filled with water which then becomes the continuous phase in the system.

Case 4, $\theta = 60$ *deg.*—A value of θ of 60 deg. represents the inverse relations of case 2. This case could be represented by the partially inverted arch, one half of which is shown in Fig. 4 (*a*), with the horizontal forces being the applied forces acting through a transmission angle of (90 deg. $-\ \theta$) and the vertical force being the resisting thrust. When θ, as measured with the vertical, is 60 deg. the horizontal pressure is three times the vertical:

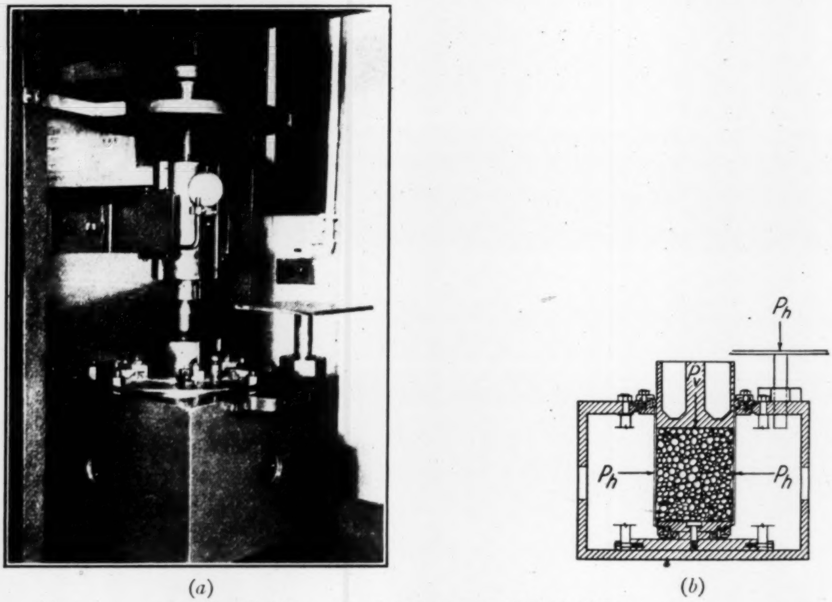
when

$$\theta = 60 \text{ deg., } \tan \theta = 1.732, \tan^2 \theta = 3, \text{ and } \cot^2 \theta = \tfrac{1}{3}$$

therefore

$$p_h = 3p_v \text{ and } p_v = \tfrac{1}{3}\, p_h$$

Case 5, $\theta = 90$ deg.—When the angle θ becomes 90 deg. the stability is zero and the mechanical arrangement is that which is approached by the loose volume of a granular mass. In terms of a uniform size of spherical particles the spheres would be arranged one on top of another in vertical lines, all of which would be in a state of unstable equilibrium and incapable of carrying any vertical pressure. It is difficult to differentiate between case 1 and case 5, one being the inverse of the other. In terms of internal stability as defined, both cases would represent unstable equilibrium as far as arch action is concerned. With the introduction of cohesion, however, as an independent factor, a zero value of θ, the angle of transmission, serves to interpret the effect of cohesion.



(*a*) (*b*)

FIG. 5.—Stabilometer for Measuring Principal Pressures.

Stabilometer Tests:

A special test procedure was developed to measure the relation between the principal pressures for various granular materials. Cylindrical specimens, vibrated to a specific density close to maximum compaction, were tested to failure under vertical load in a pressure tank in which it was possible to maintain a constant lateral pressure on the specimen. The apparatus is shown in Fig. 5. Figure 5 (*a*) shows the pressure tank assembled ready for testing a sample, and (*b*) is a line diagram illustrating the test conditions, the sample being placed in the pressure tank and protected by a rubber membrane.

The stabilometer test here described is similar to that devised by Hveem of the California State Highway Department[11] with two important exceptions. In the first place, the sample is free to expand under a constant lateral pressure and is thus capable of developing its true resistance under the action of vertical and lateral pressure. In the Hveem stabilometer and some other devices that have been used to measure the relation between the principal pressures, the sample is more or less rigidly confined and is not allowed to develop its inherent resistance to displacement under the assumed conditions. In the second place, provision has been made to test specimens with a larger ratio of height, h, to diameter, b, in order to eliminate direct transfer of load from the piston to the base plate. In the case of the present tests it was found that the h/b ratios used, although approaching the proper values, did not entirely eliminate the dimensional effects noted.

In Fig. 6 are shown the results of tests to measure the relation between a constant lateral pressure and the vertical pressure necessary to cause failure of the specimen. Each of the points shown represents the average of tests run at a specific value of the lateral pressure. Results are given for five different materials at the per cent of voids indicated which was the minimum obtainable with the method of vibration used. The h/b ratio was constant and equal to 1.30.

The results indicate essentially a constant relation between the principal pressures at the point of failure except for very low values of the lateral pressure. There is evidence from a slight curvature in the lower range of additional resistance to failure under vertical pressure which may be due to interlocking of particles causing a steeper angle of pressure transmission than is later evident. Such an effect is similar to cohesion but is more probably the result of mechanical advantage which requires very little horizontal thrust to develop. This is most evident in the angular particles with a steeper angle of pressure transmission and is not apparent in the tests on lead shot. There is some reason to believe that a contributing factor in this case is the direct transfer of load to the base plate due to the use of an h/b ratio of 1.3 which is less than the cotangent of the angle of pressure transmission. In order to eliminate this effect, such tests should be made on specimens with an h/b ratio at least equal to the cotangent of the angle of pressure transmission to avoid lateral restraint at the ends.

Shear Tests:

As a part of the investigation of granular materials the attempt was made to correlate internal friction or resistance to sliding on an inclined plane with the relation between the principal pressures. It is, of course,

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[11] T. E. Stanton and F. N. Hveem, "Role of the Laboratory in the Preliminary Investigation and Control of Materials for Low Cost Bituminous Pavements," *Proceedings*, Fourteenth Annual Meeting, Highway Research Board, Vol. 14, Part II, December, 1934.

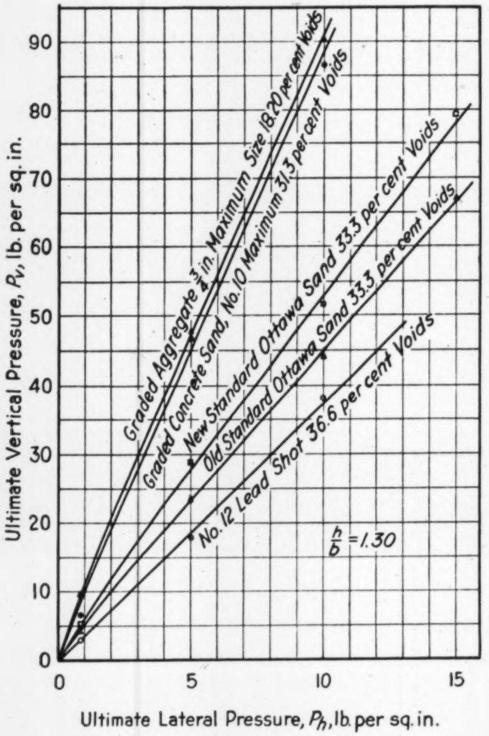

FIG. 6.—Stabilometer Test Results.

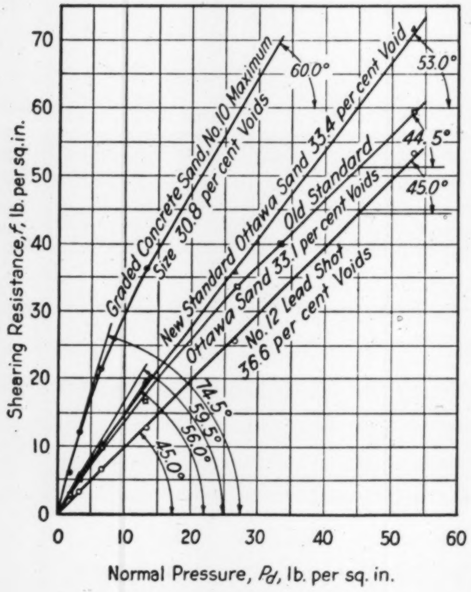

FIG. 7.—Shear Test Results.

possible to interpret the stabilometer tests in terms of a hypothetical angle of internal friction based on the assumption that the difference between the greatest and least principal pressure is controlled by resistance to sliding on a plane of minimum resistance. It must be further assumed that the ratio of the tangential stress component divided by the normal pressure on the plane of sliding is equal to the tangent of the angle of internal friction. In this investigation, tests were made to determine the angle of internal friction by a direct shear test which measured the normal and tangential components on the plane of failure.

The apparatus for the direct shear tests has been described by Berry[4] and preliminary test results presented and discussed. The author will not duplicate the description of apparatus and will present only those results pertinent to the present discussion. The shear test results for four of the materials used in the stabilometer tests is shown in Fig. 7.

No shear tests were run on Material No. 1, the graded aggregate of $\frac{3}{4}$-in. maximum size, because the shear cylinder could not be used for such large particles without introducing dimensional effects which would destroy the value of the tests. Tests on the other materials were conducted at maximum compaction with the percentage of voids essentially the same as for the stabilometer tests. The angles of friction as determined from the direct measurement of stress components on the shearing surface are shown on the figure. Maximum and minimum values of the angle are shown. As was the case in the stabilometer tests, there is evidence of an additional resistance developed in the lower range of normal pressure which is not directly proportional to the normal pressure. This could be interpreted as an interlocking of particles which requires smaller increments of normal pressure to develop. This behavior is particularly evident in the angular particles and is not apparent in the lead shot.

TABLE I.—DATA FROM STABILOMETER AND SHEAR TESTS.

Material	Stabilometer Tests					Shear Tests		
	$p_h/p_v = \tan^2\theta$	$p_v/p_h = \cot^2\theta$	$\tan\theta$	Angle θ, deg.	Angle $\phi = 90-2\theta$ deg.	Angle ϕ', deg.		Angle ϕ', $\tan^{-1} = \frac{1}{3}\cot^2\theta$, deg.
						Max.	Min.	
Graded aggregate, $\frac{3}{4}$ in. max. size	0.113	8.85	0.336	18.6	52.8	.....	....	.....
Graded concrete sand, No. 10 max. size	0.114	8.77	0.338	18.7	52.6	74.5	60	71.1
New standard Ottawa sand	0.200	5.00	0.447	24.1	41.8	59.5	53.0	59.0
Old standard Ottawa sand	0.229	4.37	0.478	25.5	39.0	56.0	44.5	55.5
No. 12 lead shot	0.267	3.75	0.517	27.3	35.4	45.0	45.0	51.3

The result of primary importance in shear tests on compacted material was that the angles of friction were all greater than 45 deg. with the exception of the lead shot which was approximately equal to 45 deg. The coefficient of friction being equal to the tangent of the angle, was then in all cases equal to or greater than unity. Similar results have been obtained in several cases by other investigators.[12] The significance of this result will be considered in detail in connection with a discussion of bearing-capacity tests.

Correlation of Shear Tests and Stabilometer Tests:

The essential data from the shear tests and stabilometer tests on the various materials are assembled in Table I for the purpose of correlation and to assist in a comparison of the proposed "arch theory" of stability with the conventional internal friction theory.

The second column is the ratio of lateral pressure to vertical pressure which is equal to the $\tan^2 \theta$ from Eq. 1 and represents the relative magnitude of the minimum principal pressure. The third column gives the inverse ratio equal to $\cot^2 \theta$ and indicates the relative magnitude of the maximum principal stress or passive resistance of the material. The fourth and fifth columns show the values of $\tan \theta$ and θ, the angle of pressure transmission, respectively. The angle of pressure transmission, as previously stated, represents the direction of motion of the granular particles when the supporting horizontal thrust is exceeded or removed. It should then be comparable to the angle of minimum resistance or plane of failure as deduced from the internal friction theory. In the sixth column the angle ϕ has been computed from these relations.

The equation for the ratio of lateral pressure to vertical pressure from Coulomb is as follows:

$$\frac{p_h}{p_v} = \tan^2\left(45 \text{ deg.} - \frac{\phi}{2}\right)$$

A comparison with Eq. 1 developed in the present discussion shows that

$$\theta = 45 - \frac{\phi}{2}$$

Then

$$\phi = 90 - 2\theta$$

A comparison of the theoretical values of ϕ, the angle of internal friction, as given in the sixth column, with the values of ϕ', measured from the shear tests in the seventh and eighth columns, shows a wide disparity. The disagreement is substantially greater for those materials which have steeper angles of pressure transmission as derived from the greater differences between the maximum and minimum principal pressures. Whether or not there is any relation between the theoretical angle of internal friction, as deduced from the Coulomb equation for principal pressures, and the angles as determined from the measurement of actual normal and tangential components or a plane of shear is a question which deserves serious consideration.

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[12] William Cain, "Experiments on Retaining Walls and Pressures on Tunnels," *Transactions*, Am. Soc. Civil Engrs., Vol. 74, p. 403 (1911).
P. M. Crosthwaite, "Experiments on Earth Pressure," *Proceedings*, Inst. Civil Engrs., Vol. CCIII, Part 2, p. 124 (1916); Vol. CCIX, p. 252 (1920).

The author believes that the evidence indicates that resistance to sliding on some plane within the granular mass is not a controlling factor in the relation between the principal pressures which is the fundamental expression of internal stability. It appears, however, that the angle of pressure transmission as conceived in the stability of the elementary soil arch is a logical and consistent means of describing internal stability and avoids the inconsistencies which have been encountered.

On the other hand, there may be some relation between the angle of pressure transmission θ and the angle ϕ' as measured by the ratio of shear to normal pressure. If there is such a relation it should be dependent on mechanical arrangement and can possibly be described in terms of static reactions between particles. In the last column of Table I an equation is suggested which appears to express the angle of internal friction ϕ' determined from the shear tests in terms of the angle of pressure transmission θ measured by the stabilometer. This angle should not be confused with the theoretical angle ϕ shown in the sixth column, as the indicated relation is independent of the principal pressures. The author is not prepared to present a complete development of the equation which is as follows:

$$\tan \phi' = \frac{1}{3} \cot^2 \theta \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

Considering this equation as empirical, it is noted in the last column of Table I that the values of ϕ', computed from the equation, compare very favorably with the maximum values of ϕ' measured by the shear test data in Fig. 7. The maximum values of ϕ' are representative of test data for normal pressures from 0 to 10 lb. per sq. in., so may logically be compared with values of θ which are determined in the stabilometer test for a range of lateral pressure from 5 to 10 lb. per sq. in.

In connection with the suggested relation, and in the absence of a rational development, it may be stated that the factor $\frac{1}{3}$ appears to be connected with the elementary soil arch of three point support and $\cot^2 \theta$ represents the passive resistance. The expression of this resistance as a shearing force involves some aspects of reorientation of particles and other relations which are not yet clear. This problem is not, however, of primary importance in the light of the conclusion that shearing resistance is not necessary to a complete description of internal stability and it may be postponed until it has been given further study.

Effect of Cohesion:

The introduction of cohesion into a granular material is necessary to a complete treatment of internal resistance of soil mixtures commonly encountered in practice and is also a part of the rational treatment of various stabilized mixtures used in highway construction. Shearing resistance may be introduced as a separate factor affecting the difference between the greatest and least principal pressure and then may be properly combined with internal stability due to arching action. The shearing stress s on a diagonal plane inclined at an angle θ with the vertical principal plane is given by the following equation:

$$s = \tfrac{1}{2} (p_v - p_h) \sin 2\theta$$

Given a specific value of the shearing resistance m', the greatest difference between the maximum and minimum principal pressure which may be maintained is given by the equation:

$$p_v - p_h = 2m'/\sin 2\theta$$

When there is a specific relation between the principal pressures controlled by the angle of pressure transmission and independent of cohesion, the two separate effects may be combined as shown in Fig. 8.

The value of vertical pressure is given by the following equation:

$$p_v = p_h \cot^2 \theta + 2m'/\sin 2\theta \; \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

The vertical pressure p_v may have any value up to $2m'/\sin 2\theta$ without requiring any lateral pressure or horizontal thrust for equilibrium. The term $p_h \cot^2 \theta$ becomes zero for smaller values of p_v and indicates that the lateral pressure is zero when the cot θ has any finite value. The significance of various values of θ has already been discussed, but it may be repeated that for θ equal to 45 deg. Eq. 6 reduces to the form $p_v - p_h = 2m'$, which is the relation for a true cohesive material without arching action. It should also be pointed out that a value of θ of 90 deg. and $\cot^2 \theta$ equal to zero leads to the same deduction and cohesion becomes the only source of internal resistance. Similar interpretations may be made from the equation for lateral pressure which is as follows:

$$p_h = (p_v - 2m'/\sin 2\theta) \tan^2 \theta \; \ldots\ldots\ldots\ldots\ldots\ldots (7)$$

Inasmuch as p_h cannot have negative values, zero lateral pressure represents a limiting case for which $\tan^2 \theta$ and θ must be zero. This is the inverse case of θ equal to 90 deg. although both reduce to the form of $p_v - p_h = 2m'$.

Two practical examples of pressure components in materials which exhibit combined arching action and cohesion are given in Fig. 9.

Case A is a well-compacted oil-aggregate mixture with a graded gravel of $\frac{3}{4}$-in. maximum size and a heavy bituminous road oil. The voids in the compacted mixture were 8.3 per cent while the voids in the aggregate, air voids plus volume of bituminous material, were approximately 16 per cent.

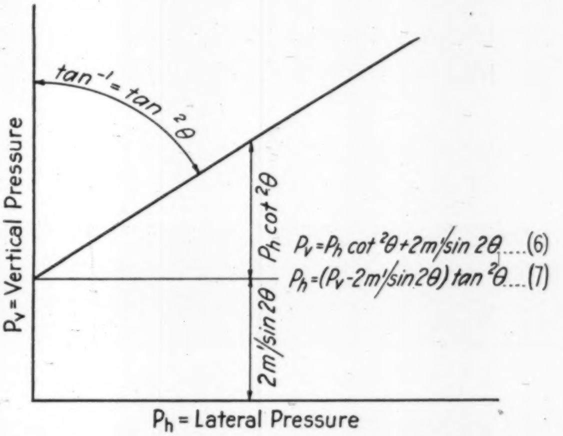
$\tan^{-1} = \tan^2\theta$
$P_h \cot^2\theta$
$2m'/\sin 2\theta$
$P_v = P_h \cot^2\theta + 2m'/\sin 2\theta$ ....(6)
$P_h = (P_v - 2m'/\sin 2\theta)\tan^2\theta$ ....(7)
P_v = Vertical Pressure
P_h = Lateral Pressure

FIG. 8.—Combination of Arching Action and Cohesion.

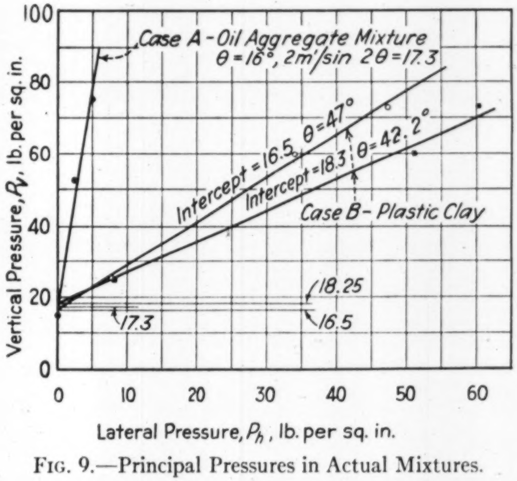

FIG. 9.—Principal Pressures in Actual Mixtures.

The tests were conducted in the stabilometer at lateral pressures of 0, 2.5, and 5 lb. per sq. in. At zero lateral pressure the specimen carried a vertical pressure of 17.3 lb. per sq. in. which, being equal to $2m'/\sin 2\theta$, represents the influence of cohesion on the inclined plane of failure. The angle of pressure transmission θ of 16 deg. is somewhat steeper than for the graded aggregate used in the previous stabilometer tests as might be expected from the smaller percentage of voids. The ratio of height to diameter of the specimen was 0.70 so that part of the high resistance indicated is unquestionably due to direct transfer of load from the piston to the base plate.

Case B is an analysis of earth pressure measurements on a vertical shaft at the Delray Power Plant of the Detroit Edison Co. Goldbeck pressure cells were installed at three different elevations and measurements have been carried on over a period of several years. There are two analyses shown representing the average of readings taken at the end of different periods of time. The shaft is a vertical caisson 4.5 ft. in diameter through a very plastic clay in which the voids are filled with water. The two sets of results indicate some variation in pressure, typical of shifting ground, and has been characteristic of the soil behavior since installation of the cells. The pressure varies with the depth at a rate approximately equal to the static pressure of a fluid weighing 125 lb. per cu. ft. This behavior is equivalent to an angle of pressure transmission of 45 deg. as pointed out in the previous discussion as typical of a clay dispersed in water. The results of two sets of data shown indicate an angle θ of 42.2 deg. and 47 deg. with intercepts on the vertical axis of 18.3 and 16.5 lb. per sq. in., respectively. These intercepts are not in this case equal to $2m'/\sin 2\theta$ as they are also affected by the diameter of the shaft and the tendency of the soil to arch around the shaft. Such dimensional effects encountered in ordinary excavation are not properly a part of this discussion and are only mentioned to avoid misinterpretation.

BEARING CAPACITY OF GRANULAR MASSES

The ultimate objective of any theory of internal resistance of soils or granular mixtures, aside from the problem of earth pressures, is to develop a rational method of defining and measuring bearing capacity or ability to support loads. While existing conceptions of internal friction have not been wholly inadequate in the treatment of earth pressures or even in describing pressure distribution, they have failed quite completely to provide a basis for solution of the problem of bearing capacity. They do not provide any means by which to evaluate boundary effects which cause a variation in the supporting power of a granular mass for different sizes of bearing area, a fact which has been well established by ample experimental evidence. Neither do they provide a logical means of describing the manner in which failure takes place when a granular material is overloaded.

Failure Under Surface Loads:

It is in the latter case that the inconsistency of the internal friction theories become most apparent in the light of recent data. Attention already has been called to the fact that, in the present investigation and others, values of the angle of friction greater than the theoretical value ϕ

have been obtained. It is evident that if there is any static resistance available on the critical plane of failure in excess of that deduced from the internal friction theory the resistance available will always be greater than the shearing stress. It must be concluded, then, that if frictional resistance is the controlling factor, granular material would have infinite bearing capacity up to the crushing strength of the particles themselves. This is obviously a false conclusion as granular materials do fail under surface loads and, in fact, may have very little bearing capacity if not well compacted. The failure universally observed is due to lateral displacement, as illustrated in Fig. 10.

In this illustration,[13] the particles beneath the bearing area move in a downward-lateral direction, and outside of the central column move in an upward-lateral direction. Lines have been drawn on the photograph to distinguish various portions of the mass. The central column is shown to divide the region where motion changes from downward to upward, the motion being practically horizontal at the planes through the edge of the bearing area. Immediately beneath the area is a penetrating wedge or keystone which moves downward due to lack of supporting horizontal thrust from the elements adjacent to the central column. There is also an inverted wedge of material which meets the penetrating wedge and is held in place by the greater horizontal thrust available at greater depths where there is little if any lateral motion.

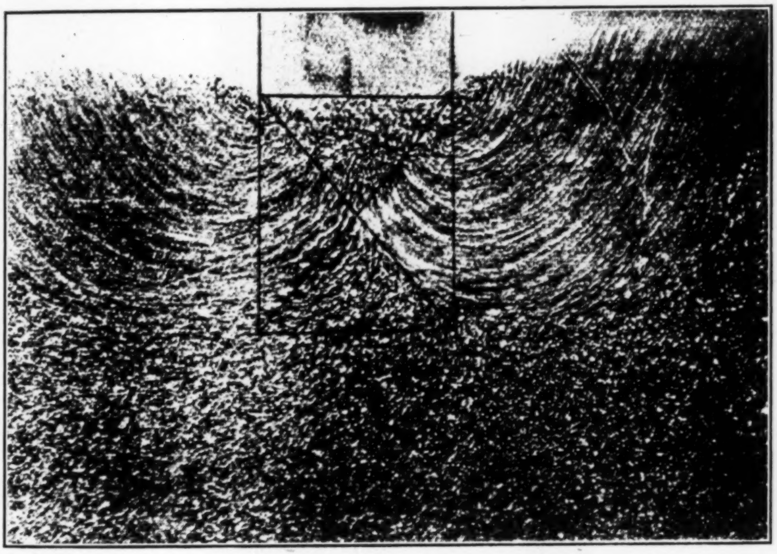

FIG. 10.—Failure of Granular Material.

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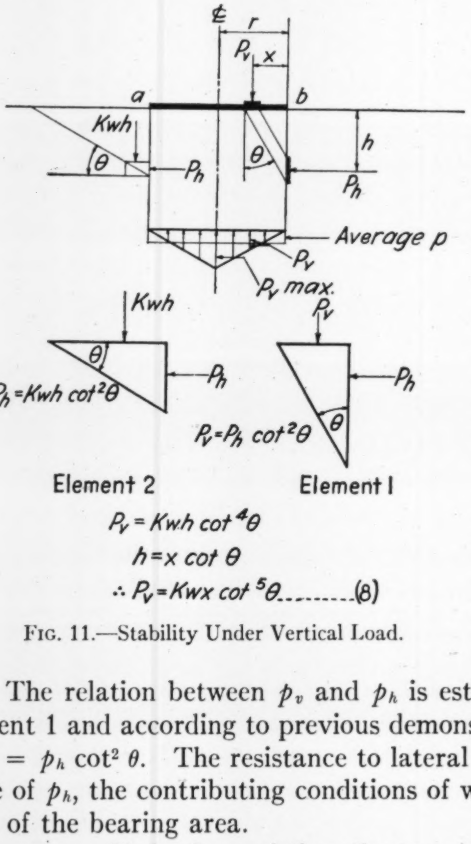
[13] Discussion by M. L. Enger of paper by G. Paaswell, "Transmission of Pressure Through Solids and Soils," *Transactions*, Am. Soc. Civil Engrs., Vol. 85, p. 1581 (1922).

Development of Equation for Ultimate Bearing Capacity:

It is possible to express the equilibrium of the various elements involved in such a failure in terms of internal stability as embodied in the conceptions of arching action developed in the preceding discussion. Figure 11 is a diagram illustrating the various factors to be considered in such a development.

There is shown a bearing area, *ab*, of radius, *r*, resting on the horizontal free surface of a granular mass. At a distance x from the edge of the bearing area the pressure p_v is supported by the horizontal thrust p_h with the resultant force acting along the arch axis which makes an angle θ with the vertical. The relation between p_v and p_h is established by the equilibrium of element 1 and according to previous demonstration is given by the equation $p_v = p_h \cot^2 \theta$. The resistance to lateral movement controls the magnitude of p_h, the contributing conditions of which are shown under the left edge of the bearing area.



℄
r
P_v
x
a
b
Kwh
θ
P_h
θ
h
P_h
Average p
P_v
P_v max.
Kwh
θ
P_h
$P_h = Kwh \cot^2\theta$
P_v
P_h
$P_v = P_h \cot^2\theta$
θ
Element 2
Element 1
$P_v = Kwh \cot^4\theta$
$h = x \cot\theta$
$\therefore P_v = Kwx \cot^5\theta$ ....(8)

FIG. 11.—Stability Under Vertical Load.

The vertical pressure, *Kwh*, the resisting thrust against which the lateral pressure of p_h acts, arises from two sources. In the first place, the weight of the material is given by *wh* and varies from zero at the surface in direct proportion to the depth. The factor, *K*, represents vertical pressure transmitted to the elements outside the central column due to lateral distribution of pressure from the bearing area. This vertical pressure is also zero at the surface but varies according to laws which have been established in connection with the distribution of vertical pressure from concentrated loads. This factor is so related to boundary conditions for different sizes of bearing area that it must be determined from actual load tests and enters the equation for bearing capacity as an independent variable.

The magnitude of horizontal thrust, p_h, available in the development of bearing capacity, is determined by the equation, $p_h = Kwh \cot^2 \theta$, from the equilibrium of element 2. The combined resistance available from both elements is given by the equations:

$$p_v = Kwh \cot^4 \theta$$

$$h = x \cot \theta$$

then

$$p_v = Kwx \cot^5 \theta \quad \text{(8)}$$

The ultimate vertical pressure, p_v, developed by arching action, varies from zero at the edge of the plate where $x = 0$ to maximum at the center where $x = r$. The pressure distribution at the ultimate bearing capacity resulting from the development given is represented by a triangular variation suggested by Kogler and Scheidig[14] as a suitable approximation for the more complex pressure distribution curves which result from the mathematical treatment of pressure distribution. This distribution holds only at the ultimate bearing capacity and must be altered for loads less than the ultimate.

For practical purposes it is essential to express the bearing capacity in terms of the average pressure over the area, which in this case will be assumed to be circular. The value of the maximum pressure, p_v, at the center of a round bearing area is obtained from Eq. 8 for a value of $x = r$ and is as follows:

$$p_v \max = Kwr \cot^5 \theta \quad \text{(9)}$$

The equation for bearing capacity in the usual terms of average pressure is obtained by equating the volume of the cone of pressure with an altitude of p_v maximum to the volume of a cylinder with a height equal to p, the average pressure. The equation for ultimate bearing capacity is then:

$$p = \tfrac{1}{3} Kwr \cot^5 \theta \quad \text{(10)}$$

Analysis of Bearing-Capacity Tests:

Series of bearing-capacity tests were conducted on each of the five materials used in the stabilometer and shear tests previously described. The analysis of these tests by Eq. 10 is shown in Table II.

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[14] F. Kogler and A. Scheidig, "Pressure Distribution in Building Soil," *Die Bautechnik*, No. 18, March 17, 1929.

The only unknown in Eq. 10 is the factor K, which measures the resistance developed by pressures transmitted from the bearing area to elements outside of the central column. If the equation properly describes the measured bearing capacity, it should be constant for various sizes of the bearing area. The computed values of K are shown in Table II for each size of bearing area and the average value for each material is also computed. The values of K shown are remarkably consistent with the possible exception of the tests on the 4 sq. in. bearing area for graded aggregate of $\frac{3}{4}$-in. maximum size. This particular test gives higher values for ultimate bearing capacity than might be anticipated from analysis of the tests on the 8 and 16 sq. in. areas.

TABLE II.—DATA FROM BEARING-CAPACITY TESTS.

Material	Depth of Sample, in.	Angle of Pressure Transmission, θ, deg.	$\cot^5 \theta$	Volume Weight, w, lb. per cu. in.	Area, sq. in.	Radius, in.	wr, $\cot^5 \theta$	Ultimate Bearing Capacity, p, lb. per sq. in.	$K = \frac{3p}{wr \cot^5 \theta}$	Computed Bearing Capacity, $p = \frac{Kwr \cot^5 \theta}{3}$, lb. per sq. in.
Graded aggregate, $\frac{3}{4}$-in. max. size.	5.0	18.6	235	0.0782	4	1.128	20.70	130	18.83	114.0
					8	1.596	29.35	152	15.53	161.5
					16	2.258	41.50	210	15.19	228.0
								Av. K	16.52	
Graded concrete sand, No. 10 max. size....................	6.1	18.7	228	0.0657	4	1.128	16.90	63	11.18	64.5
					8	1.596	23.90	91	11.42	91.1
					16	2.258	33.80	131.5	11.67	128.9
								Av. K	11.42	
New standard Ottawa sand.....	4.0	24.1	56	0.0638	4	1.128	4.53	21	13.92	22.4
					8	1.596	5.70	29.4	15.48	28.2
					16	2.258	8.05	40.6	15.12	39.8
								Av. K	14.84	
Old standard Ottawa sand......	6.4	25.5	39.6	0.0638	4	1.128	2.85	13.25	13.95	12.4
					8	1.596	4.03	17.15	12.77	17.52
					16	2.258	5.70	23.50	12.37	24.80
					32	3.194	8.06	35.25	13.12	35.05
								Av K	13.05	
No. 12 lead shot...............	4.0	27.3	27.2	0.260	4	1.128	7.97	2.50	0.941	2.34
					8	1.596	11.28	3.22	0.857	3.32
					16	2.258	15.95	4.50	0.846	4.69
								Av. K	0.881	

The average value of K for each material has been used to compute the ultimate bearing capacity for the different sizes of bearing areas. The computed capacities are shown in the last column of Table II. This affords the most direct comparison with the measured values in the ninth column. With the exception noted above, the close agreement indicates that Eq. 10

as developed is an accurate representation of actual bearing-capacity tests. It also indicates that the angle of pressure transmission θ as determined by the stabilometer tests has been properly evaluated as a controlling factor in the stability of granular materials.

Effect of Surcharge:

With the assurance of experimental evidence that the rational development of Eq. 10 has been properly carried out, there are a number of generalizations to be made and several other factors to be considered in a complete treatment of internal stability. The first of these is the increment of bearing capacity due to surcharge. Equation 10, as well as the bearing-capacity tests analyzed, refer to load applications on the free surface of a granular mass. If a surcharge equivalent to a static head of h_1 is introduced on the free surface, the bearing capacity made available may be deduced directly from the relations shown on Fig. 11. The vertical pressure acting on element 2 is increased by an amount equal to wh_1 which is constant at all depths so h_1 is independent of x, the variable distance from the edge of the bearing area measured toward the center of the area. In other words, the surcharge adds a constant pressure ordinate to the bearing capacity which is independent of the size of the area and is given by the equation:

$$p = wh_1 \cot^4 \theta$$

This factor may be added directly to Eq. 10 which then becomes a general equation for bearing capacity of a granular material with surcharge. The equation is as follows:

$$p = \tfrac{1}{3} Kwr \cot^5 \theta + wh_1 \cot^4 \theta \ldots\ldots\ldots\ldots\ldots\ldots (11)$$

Effect of Cohesion:

The combination of cohesion with arching action has been indicated on Fig. 8 in the discussion of principal pressures and amounts simply to an increased difference between the greatest and least principal stress. Referring to Fig. 11, shearing resistance enters the development by increasing the maximum principal pressure p_v acting on element 1 by an amount equal to $2m'/\sin 2\theta$. In a similar manner the maximum principal pressure p_h acting on element 2 is increased by the addition of $2m'/\sin 2\theta$. The general equation for ultimate bearing capacity with a combination of arching action, cohesion, and surcharge is then as follows:

$$p = \tfrac{1}{3} Kwr \cot^5 \theta + wh_1 \cot^4 \theta + 4m'/\sin 2\theta \ldots\ldots\ldots (12)$$

Bearing-capacity tests with a combination of all three factors are not available, but the factors have been separately measured by tests which may be interpreted by Eq. 12. A number of series of bearing-capacity tests on cohesive soil, in some cases with surcharge, have been made by the author on plastic clay which would correspond to a value of θ equal to 45 deg. The correlation of shearing resistance, m', with bearing capacity has been published.[15] In the correlation of these tests in which the relation $4m'$ has been demonstrated, and for cases where confined tests with surcharge were made, the factor of static head was found to add to the developed pressure an amount equal to wh_1. In Eq. 12, the introduction of a value of θ equal to 45 deg. reduces the surcharge effect to this amount and so constitutes a partial verification of the correctness of the surcharge term. Aside from this, the treatment of surcharge must rest upon the rational development of the general Eq. 12.

Methods of Evaluating Variable Factors:

It is pertinent, at this point in the discussion, to review the methods available for measuring the various factors that have been isolated in the complete equation for bearing capacity. It is also pertinent to point out that these factors are subject to direct measurement in order to demonstrate that the theory developed deals with observable magnitudes.

Two of the quantities, w and h_1, obviously present no difficulties in measurement. The static head, h_1, is simply a test condition which is easily determined. The weight per unit of volume can be accurately measured in the laboratory and requires only that care be exercised in controlling the density so that it be representative of the state of compaction for which the bearing capacity is to be measured.

The shearing resistance m' may be measured by the stabilometer test when properly separated from internal stability due to arching action, as indicated in Figs. 8 and 9. The intercept on the vertical axis gives the cohesion factor directly and may be a measure of actual shearing resistance dependent on a proper analysis of the test conditions. In the case of strictly cohesive materials, such as clay, methods have been developed for the direct measurement of shear.[15]

The angle of pressure transmission, θ, is a factor of primary importance in the theory of stability that has been developed. The stabilometer test, which has been described in detail, measures this angle directly as a function of the difference between the principal pressures. The correlation of experimental data, which also has been discussed, is evidence not only that the test is a proper one, but that it is adaptable to the problems encountered in practical testing procedure.

It has also been pointed out that the angle of pressure transmission is analogous to the angle of repose, provided the test conditions are properly controlled. It is recognized that the natural slope which a granular mass will assume over a long period of time will not represent the angle of pressure transmission in the interior of the mass. If, however, a specimen is properly prepared at a specific density requiring a specific mechanical arrangement, it appears that the angle of repose assumed upon removal of the supporting horizontal thrust may be representative of the angle of pressure transmission. It is obvious, for instance, that in a pyramid of spheres with regular arrangement representative of maximum density, the angle of repose and the angle of pressure transmission are the same. If these conditions can be duplicated in other granular materials, such a simple procedure would be an extremely practical test and may be of sufficient accuracy for many purposes.

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[15] W. S. Housel, "A Penetration Method for Measuring Soil Resistance," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 472 (1935).

In so far as bearing capacity is concerned, the factor K is the only variable that cannot be measured by independent tests. It depends fundamentally on boundary effects which cannot be duplicated in any way and involves conditions which are not now, and may never be, subject to analysis. It may be measured by bearing-capacity tests as in the present analysis, but other possibilities appear rather vague at the present time. It may be possible that K is so related to the angle of pressure transmission, θ, that after sufficient bearing-capacity data are available, an empirical relation can be established. If this is accomplished there may be specific values of K for different materials and different angles of pressure transmission. Further investigation and study alone will determine this possibility.

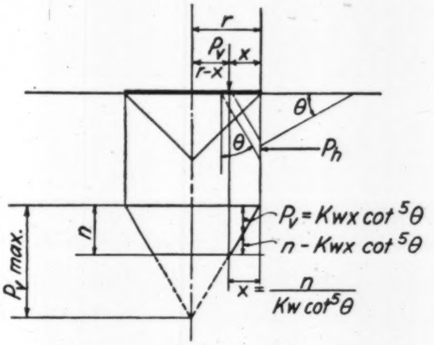

FIG. 12.—Pressure Distribution for Loads Less than Ultimate.

General Equation for Bearing Capacity:

Due to the number of factors which have been introduced in the development of a theory of bearing capacity it appears desirable to generalize Eq. 12 by combining those factors which affect bearing capacity in a similar manner. This is also necessary in order to represent bearing capacity at loads less than the ultimate which involves some variations more complex than those included in Eq. 12. The basis for the development of a more general equation is shown in Fig. 12 which represents the pressure distribution on a bearing area for loads less than ultimate bearing capacity.

In Fig. 12 is shown a bearing area of radius r in which a pressure, p_v, is acting a distance x from the edge. The relation between p_v and the lateral thrust, p_h, has been previously established, but in this case it is assumed that the applied load is such that the lateral thrusts at a greater depth than p_h are sufficient to support the applied vertical pressure without lateral displacement. The pressure on the bearing area would then vary from zero at the edge to p_v at a distance x from the edge. For greater distances from the edge there would be a uniform pressure, n, equal to the applied pressure. As shown, the pressure distribution is made up of a uniform pressure, n, and negative boundary effect due to loss of stability for the specified depth, which is represented by the ordinate:

$$n - Kwx \cot^5 \theta$$

The critical value of x occurs at the change in type of pressure distribution when the negative boundary effect is equal to zero.

$$n - Kwx \cot^5 \theta = 0$$

and

$$x = \frac{n}{Kw \cot^5 \theta}$$

The average pressure or bearing capacity, p, is derived by equating the volume of the truncated cone of pressure to the volume of a cylinder with a height equal to the average pressure, p. The development of the necessary equations follows:

$$\text{Volume of truncated cone} = \frac{\pi n}{3} [r^2 + r(r - x) + (r - x)^2]$$

$$= \frac{\pi n}{3} (3r^2 - 3rx + x^2)$$

$$\text{Volume of cylinder} = \pi r^2 p$$

$$p = \frac{n}{3r^2} (3r^2 - 3rx + x^2)$$

If the effects of surcharge and cohesion, which are both independent of x, are ignored for the moment, the interpretation of the equation for different values of x may be more clearly presented.

When $x = o$

$$p = n = o.$$

When $x = r$

$$p = \frac{n}{3} = \frac{p_v \max}{3} = \tfrac{1}{3}\, Kwr \cot^5 \theta \ldots\ldots\ldots\ldots\ldots (10)$$

When $x = \dfrac{n}{Kw \cot^5\theta}$

$$p = \frac{n}{3r^2}\left[3r^2 - \frac{3rn}{Kw \cot^5 \theta} + \frac{n^2}{(Kw \cot^5 \theta)^2}\right]$$

$$p = n - \frac{n^2}{Kwr \cot^5 \theta} + \frac{n^3}{3\,(Kwr \cot^5\theta)^2}$$

Substituting the perimeter-area ratio $\dfrac{P}{A} = \dfrac{2}{r}$ for round bearing areas,

$$p = n + \frac{P}{A}\left[\frac{n^3}{6r\,(\mathrm{Kw} \cot^5 \theta)^2} - \frac{n^2}{2Kw \cot^5 \theta}\right] \ldots\ldots\ldots\ldots (13)$$

Let the coefficient of the perimeter-area ratio, which represents the effect of dimensional changes, be designated as m, which has previously been defined as a boundary stress-reaction, perimeter shear.[16]

$$m = \frac{n^3}{6r\,(Kw \cot^5\theta)^2} - \frac{n^2}{2Kw \cot^5\theta}$$

One of the terms in the equation for m still contains the radius r, so that m cannot be used to represent a boundary stress reaction which will be independent of the size of bearing area. By expressing this additional dimensional effect in terms of the perimeter-area ratio, the expression for m may be simplified:

$$m = C_3 \frac{P}{A} + C_4 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (14)$$

$$C_3 = \frac{n^3}{12\,(Kw \cot^5 \theta)^2} \text{ and } C_4 = - \frac{n^2}{2Kw \cot^5 \theta}$$

Some preliminary analyses of bearing-capacity tests for loads less than the ultimate indicate that the magnitude of the term C_3 is such that it may be neglected without serious error within the range of loads not too near the ultimate capacity. In Eq. 14 it is also apparent that for large bearing areas with small perimeter-area ratios the order of magnitude of the variable factor in the perimeter shear is again decreased. Within those limits which allow m to be constant for various sizes of bearing area, Eq. 13

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[16] W. S. Housel, "Bearing Power of Clay Is Determinable," *Engineering News-Record*, Vol. 110, February, 1933, p. 244.

may be reduced to the linear equation for bearing capacity which the author has previously used in the analysis of bearing capacity tests on cohesive soils. This equation as applied to granular material may be written in the same terms:

$$p = m\frac{P}{A} + n \qquad (15)$$

$$m = -\frac{n^2}{2Kw\cot^5\theta}$$

The application of this equation involves another reduction in the relative importance of the perimeter shear, *m*. For large bearing areas the relative importance of the perimeter shear, *m*, is itself greatly decreased so that in three successive steps the variation in *m* for different sizes of bearing area has been shown to decrease in relative importance. It is when the attempt is made to project test data from very small areas into the range of large areas that the limitations of Eq. 15 must be recognized and an extra step in the analysis is then required to evaluate the variable boundary reaction. It is also necessary to restrict the linear approximation to loads considerably less than the ultimate, which is ordinarily the practical working range in design.

Assuming that the linear equation, Eq. 15, is restricted to its proper range of application, it is of next importance to interpret the negative value of the perimeter shear, *m*. From the relations shown in Fig. 12, it may be seen that the boundary reaction is negative and represents a loss in capacity at the edge due to lateral displacement of the particles which furnish horizontal thrust and support for the vertical load near the edge of the area. This interpretation is also entirely consistent with the variation in pressure on the bearing area which is the combination of an element of uniform pressure with negative ordinates subtracted in the region near the edge of the loaded area.

General Equation for Ultimate Bearing Capacity:

While it is important in a comprehensive description of bearing capacity to consider loads less than the ultimate, it is even more important to simplify the equation for ultimate bearing capacity including all the factors involved. Equation 12, which evaluates arching action, cohesion, and surcharge may be so simplified that it may be used to interpret bearing capacity tests without relying on the independent measurement of the various factors involved:

$$p = \tfrac{1}{3} Kwr \cot^5\theta + wh_1 \cot^4\theta + 4m'/\sin 2\theta \qquad (12)$$

It already has been shown that surcharge and cohesion are independent of the size of bearing area so they may be combined in a single factor of developed pressure, n, used in the preceding derivations for loads less than the ultimate. The radius, r, may also be expressed in terms of the perimeter-area ratio and the general equation then may be expressed as follows:

$$\frac{P}{A} = \frac{2}{r}$$

Then

$$p = \frac{2\ Kw \cot^5 \theta}{3} \frac{A}{P} + n$$

$$p = \frac{C_1}{\frac{P}{A}} + n \ldots\ldots\ldots\ldots\ldots\ldots\ldots (16)$$

$$C_1 = \frac{2\ Kw \cot^5 \theta}{3} \qquad\qquad n = wh_1 \cot^4 \theta + 4m'/\sin 2\theta$$

Equation 16 is a hyperbola, which has been used successfully in the analysis of the ultimate bearing capacity of granular materials for both large and small bearing areas. In Fig. 13 the curves of ultimate capacity for the five materials compared in Table II are shown.

In order to bring the curves for materials with such a wide range of ultimate capacity together on the same graph, different scales have been used for the vertical ordinate for ultimate capacity.

It may be pointed out in connection with Eq. 16 that a variation in capacity inversely proportional to the perimeter-area ratio could be shown as a linear relation or direct proportion to the area-perimeter ratio or diameter of the bearing area. The author prefers the perimeter-area ratio as it serves to reduce boundary stress reactions to terms of an equivalent average pressure which may be included in the bearing capacity or average pressure which is the accepted measure of ability to support load. The perimeter-area ratio is also more advantageous in graphical representation of bearing capacity, as this ratio approaches zero for the very large bearing area encountered in practical design and makes it more convenient to correlate measured capacity in the range of test sizes with allowable capacities in the design range.

CONCLUSION

In this paper some emphasis has been placed on certain inconsistencies which have arisen in the development and extension of internal friction theories to problems of stability of granular masses. The objections to the orthodox theories may be summarized as follows:

1. Friction, which is characteristic of a state of motion is not a sound basis for describing static equilibrium.

2. Planes of failure deduced from internal friction are planes of minimum resistance rather than planes of maximum stress and violate the assumption of isotropy.

3. The application of internal friction theories has led to confusion of the angle of repose and the angle of internal friction, largely because the latter has not been isolated as an independent variable.

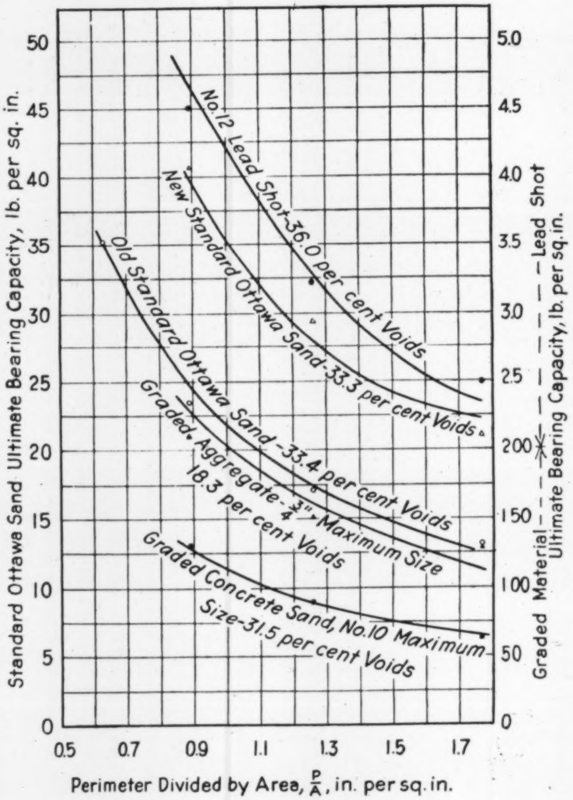

FIG. 13.—Curves of Ultimate Bearing Capacity.

4. Experimental evidence shows that actual measurement of tangential and normal components on a plane of shear gives an angle of internal friction which does not agree with the theoretical angle deduced from the measurement of principal pressures.

5. The attempt to describe bearing capacity in terms of resistance to sliding fails completely when static resistance is in excess of the theoretical internal friction, because it leads to the conclusion of infinite bearing capacity which cannot be accepted.

6. Internal friction theories fail completely in providing a basis for describing boundary conditions which control the variation in bearing capacity for different sizes of bearing areas which has been confirmed by ample experimental observations.

The author feels that the proposed "arch theory" avoids these inconsistencies and offers primary advantages in dealing with the problem of internal stability. More specifically, under the concepts of this theory it may be stated that:

1. Internal stability is described in terms of static equilibrium which avoids the necessity of assuming motion or impending motion. It may also be pointed out that so-called active and passive pressures are adequately described by a single condition of equilibrium.

2. The description of internal stability by arching action does not presuppose a condition of isotropy nor does it lead to the conclusion of a plane surface of failure. It may be concluded from the "arch theory" that when equilibrium is disturbed the direction of displacement changes after initial movement and particles will move along some curve which may or may not be determinate.

3. The angle of pressure transmission and the initial angle of repose are the same and further misinterpretation may be avoided. With proper regulation of test conditions, a simple test may be devised to measure this controlling factor in internal stability.

4. Experimental evidence shows that the angle of pressure transmission accurately describes the relation between the principal pressures in a granular mass and replaces the angle of internal friction which is shown to be independent of the principal pressures.

5. A rational development of ultimate bearing capacity has been made possible, and the mechanics of failure logically and simply portrayed in a manner consistent with observations.

6. Boundary conditions which are essential factors in the variation of bearing capacity for different sizes of bearing area have been evaluated in a general equation which interprets test data accurately.

In addition to pointing out the objections to internal friction theories which are satisfactorily overcome by the arch theory there are several other arguments which might be listed in support of the latter group of conceptions, as follows:

1. It has been shown that internal stability of the elementary soil arch is an adequate basis of describing three basic phenomena of great practical importance. These are: first, pressure distribution under load concentrations; second, the relation between principal pressures in a granular mass; and third, the bearing capacity of granular masses.

2. The various factors controlling stability have been isolated and expressed in terms of magnitudes which are subject to direct measurement.

3. Apparatus and test procedure have been developed and the correlation of data has been made which demonstrates the applicability of the theory and methods to practical problems of testing and design.

The author recognizes that a theory proposed to replace one of long standing must be subjected to critical examination before it can be accepted. It is further felt that an extension of exploratory work and the development of certain relationships are desirable. In several cases possible test methods and other developments which must await further investigation have been pointed out. A simple and practical test to measure angle of repose appears possible, but must be verified with careful corollary data. The accumulation of sufficient bearing-capacity data for granular materials may establish a relation between the dimensional factor K and the angle of pressure transmission and eliminate the necessity for a complete series of bearing-capacity tests for each material encountered. Further study is needed to establish the relation, if any, which exists between resistance to shear in a granular mass and internal stability. These problems and others that undoubtedly will arise in the extension and practical application of the proposed theory constitute a field for future research.

DISCUSSION

Mr. D. P. Krynine[1] (*presented in written form*).—The author of this paper advances an "arch theory" of internal stability in order to explain the mechanics of internal resistance of granular masses. The material of which a sand grain is made is practically incompressible; and this corresponds to a value of the reciprocal of the Poisson's ratio (Poisson number) $m = 2$. Practice has shown, however, that within a dry coarse sand mass bounded above with a horizontal plane, there is a vertical pressure, p_v, and a horizontal pressure, p_h, and that the ratio of the two is about 3 or more. Assuming this ratio equal to 3, and applying elastic formulas, one has to conclude that Poisson's ratio of the sand mass in question, considered as a whole, should equal $\frac{1}{4}$, wherefrom $m = 4$. Mr. Housel's theory explains this apparent contradiction since, according to this theory, the horizontal pressure is caused by separate grains which act as independent solids (Fig. 3), and the value of the "angle of distribution", θ, about 30 deg., is to be applied in the case referred to. If the voids of a sand mass are completely filled with water an incompressible mass (sand + water) would be formed. One could expect that both the vertical and horizontal pressure are equal in this case, since the reciprocal of the Poisson's ratio of both materials forming the mass is $m = 2$. However, the horizontal pressure, p_h, in the mass in question would be but slightly different from that in the dry sand, and in the opinion of the writer this is the proof that Mr. Housel's assumption of arch action of sand grains (Fig. 3) is correct. Mr. Housel's theory is not intended to hold in the case of clays and actually it does not; in a dispersed clay in which the voids are completely filled with water, the latter becomes the dominant phase with the tendency of the horizontal pressure to equalize the vertical. To this boundary case an angle of distribution, of $\theta = 45$ deg., would correspond in the theory of Mr. Housel.

So far as the arch phenomenon is concerned, the writer believes that it should be interpreted rather "statistically." In a photoelastic model of a sand mass prepared by Mr. D. W. Taylor, of the Soil Mechanics Laboratory, Massachusetts Institute of Technology, a great number of bakelite disks of three different diameters placed between two glasses to imitate a sand mass are compressed. In this connection stress lines going from the load to the bottom of the experimental box are being formed. These stress lines, connecting series of grains, are in the state of unstable equilibrium and change their directions until final stable equilibrium is reached. The writer is of the opinion that Mr. Taylor's model imitates the actual sand mass in a satisfactory way. Apparently when a sand mass reaches the state of stable equilibrium some grains only are stressed to a full extent, and perhaps some work but slightly. The angle of distribution, θ, is probably different in the case of individual grains; but the whole mass behaves as though it were a hypothetical mass composed of grains of equal diameter possessing a constant angle of distribution, θ.

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[1] Research Associate in Soil Mechanics, Department of Civil Engineering, Yale University, New Haven, Conn.

Very interesting is the method applied by Mr. Housel for measuring the ratio of the principal stresses (Fig. 5). Russian engineers use for this purpose a device in which the horizontal pressure exerted by the compressed soil sample on the surrounding liquid is measured with a manometer.[2] In Japan the liquid surrounding the sample is connected with a narrow pipe. Fluctuations of the level in the pipe permit the computation of changes in volume of the sample and hence the Poisson ratio.[3] Concerning Fig. 9 (Case B—plastic clay) it would be very interesting to check this test in the light of the new theories recently advanced by Terzaghi.[4]

Mr. Housel has dedicated a great portion of his paper to the problem of the bearing capacity of granular masses. His interpretation of the failure of a granular mass is not based on the patterns given by elastic theories as is usually done, but on his newly advanced "arch theory." Computed and observed results check satisfactorily (Table II). Simplifications of actually used complicated theories are welcome; and from this point of view Mr. Housel's work merits every attention. In concluding, the writer wishes to say that further continuation of Mr. Housel's interesting experiments is very desirable. Both rigid and non-rigid plates of different size and shape, not only circular, should be used in experiments, and especial emphasis should be laid on the application of the theory to the design of full sized structures.

MR. I. F. MORRISON[5] (*by letter*).—In the engineering of structures, stability is of bifurcate character. On the one hand, an element of the structure, a long slender strut for example, may be of itself unstable under certain loads which are brought to it by the other members of the assemblage. This is one type of structural instability. On the other hand, the whole assemblage of members may be so arranged that the structure is either stable or unstable.

Stability of a granular mass is of the second kind. The collapse of individual grains is not considered. It has, then, to do with the configuration of the particles and the forces which hold them in place. They may be in the stable or the unstable state. An arrangement of particles is unstable when it can pass over into another configuration upon being slightly disturbed. When in the stable state, it tends to maintain its configuration against disturbances. It has become customary, when speaking of granular masses, to call these the "loose" and "dense" states respectively. Any granular mass in nature is usually in a state between these extremes.

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[2] N. Gersevanov, "Improved Methods of the Consolidation Test," *Proceedings*, Internat. Conference on Soil Mechanics and Foundation Eng., Vol. I, p. 49, Fig. 1 (1936).

[3] Reports on Soil Tests, *Bulletin*, Geotechnical Committee, Government Railways of Japan, No. 4, p. 53, Figs. 1 and 2 (1936).

[4] K. Terzaghi, "The Shearing Resistance of Saturated Soils," *Proceedings*, Internat. Conference on Soil Mechanics and Foundation Eng., Vol. I, pp. 54–56 (1936).

[5] Professor of Applied Mechanics, University of Alberta, Edmonton, Alta., Canada.

It is difficult then to comprehend the precise meaning of the title of this paper, for it has to do with experiments on masses of granular materials in the dense or stable state, for in all cases the specimens were "vibrated to a specific density close to maximum compaction." It would appear that the author is really dealing with the ultimate internal resistance offered by granular masses to change of shape and to rupture along certain surfaces.

The writer cannot agree with the author that "friction is characteristic of a state of motion." Friction is defined as that force which tends to oppose motion produced by other forces. It is not dynamic—dynamic in the restricted meaning—in character, but is intrinsically passive. Quantitatively, it is limited in any particular case. As the force tending to produce motion increases, it increases in like amount, so that up to a certain limit static equilibrium is maintained. When the threshold value is reached movement commences and the force of friction, which continues to oppose the motion, may or may not, according to circumstances, remain constant.

The physical nature of friction has its seat in the interaction of atomic forces and is closely related to adhesion. The phenomenon is intricate and complicated by the fact that there are several stages depending on the physical conditions. The coefficient of friction is constant only under certain circumstances, and even then the friction is proportional to the normal pressure between the interacting surfaces only within a certain range of magnitude of normal pressure. In considering the intergranular friction existing between the particles of an aggregate, one must visualize the tangential forces called into play at the numerous points of contact as extending over a wide range of values because of the extremely variable amount of the friction at these places depending on surface conditions. Smooth surfaces which are clean and have high pressures at their contacting points possess enormous frictional forces which are not proportional to the normal pressures between surfaces. On the other hand, rough surfaces and contaminated surfaces will have much smaller friction available to resist motion. Surfaces in nature are contaminated by adherent layers of matter and much depends, when considering friction, on the relation between these layers and the intensity of the normal pressures at small contacting regions. Coulomb's law of friction applys only in a general way to large contaminated surfaces having moderate normal pressures. There is no assurance that it can be applied to the friction existing between the small particles of a granular mass.

It would appear, therefore, that the proper conception of the frictional forces which can be brought into existence between particles should be that they have a wide range of magnitude as one passes from one contacting surface to another. The expression "internal friction" as applied to a granular mass should be reserved for this concept.

Another important feature which is associated with internal friction but which should be kept—in thought—separated from it, is the interlocking of particles. This is dependent solely on the shape of the particles. The resistance offered by interlocking, apart from the actual strength of the particles, is however, dependent on such forces as intergranular adhesion, intergranular friction, gravitation, external pressures, etc., which tend to keep the particles from becoming unlocked. Like friction it has a passive character.

By way of example, a mass of small steel balls will have practically no interlocking, and is easily deformed. On the other hand a mass of shingle nails, with the same internal friction, is practically rigid on account of the interlocking.

Internal resistance, often called internal friction, results from the combined action of intergranular friction and interlocking of particles.

It is for the above reasons that the process of abstraction must be applied to masses of granular materials in order to arrive at an idealized granular mass which can be dealt with by the usual method of stress analysis. By this process the assumption of statistical isotropy arises and once it has been made it does not appear logical to return to the concepts of a discrete system even though the particles are left endowed with uniform properties such as shape, size, inter-particle friction, etc. It is difficult, therefore, to understand the author's objection to the adoption of the classical methods and to accept the statement that "the failure plane is one of minimum resistance and does not coincide with the plane of maximum stress." If such is to be accepted, then the assumption of isotropy must be given up and this depends on the order of the case under discussion. For very small masses of granular materials—small in relation to the size of the individual particle—it is obvious that the assumption of isotropy would be unreasonable. On the other hand, for very large masses the obvious objection no longer holds. A comparison may well be made between the case in hand and the considerations now being given to the fracture of crystalline materials. Single crystals are not isotropic and the classical stress analysis does not apply, especially at the point of shearing failure. Slip takes place on a certain plane of minimum resistance which usually does not coincide with the plane of maximum shearing stress.

Regarding the matter from a microscopic point of view it may be that the rupture through a granular mass takes place by the propagation of a localized disturbance among the grains, similar to the passage of a dislocation as suggested by G. I. Taylor for the slip of crystal grains. From the macroscopic point of view the rupture must be the result of an integrated effect. In large masses, however, it hardly seems advisable to attempt to deal with individual grains or even small groups of grains any more than, with polycrystals, one attempts to deal with individual grains. The actual process of rupture is still obscure and it will be very difficult to arrive at a theory of ultimate bearing capacity based on the microscopic point of view until that process is fully understood.

The experimental results shown in Figs. 6 and 7 are for granular materials in a dense state at the instant of failure. The effect of interlocking of particles is revealed by the steeper lines for the more angular grained materials. There appears to be no way in which the effect of intergranular friction can be separated from the effect of intergranular locking. The latter effect can merely be recognized as an important factor in the phenomenon of internal resistance. Moreover, accepting the author's definition of the "angle of pressure transmission," they show that this angle is nearly constant when the point of failure is reached for varying vertical and lateral pressures within the range of pressures used.

There is nothing in the experimental results which shows the relation between the angle of pressure transmission and the applied vertical pressure before failure takes place. The writer wishes to raise the point that the angle of pressure transmission may be a function of the magnitude of the vertical load. If this is so, then the principle of superposition is not applicable, at least without the reservations.

No mention is made of the moisture content of the materials used and it is presumed, therefore, that they were in the air-dried condition. The effect of moisture is to resist compaction up to a certain percentage of water content, after which it aids compaction making it possible to obtain a more dense mass than is possible in the dry state. At some rather well-defined percentage of moisture, the maximum density is reached. Further addition of moisture again decreases the density. This phenomenon is dependent primarily on the particle shape. It does not appear for spherical particles. It would seem, therefore, that the angle of pressure transmission depends on the extent of compaction of the mass and therefore also on the moisture content.

The writer does not wish to enter into a discussion of the development of the equation for ultimate bearing capacity but would point out that it is difficult to see why the two sources from which the resisting thrust arises, and which appear to be quite independent, should be combined in a single term in the equation for resistance to lateral movement. That no discrepancy due to this coalescence of terms appears in the results of Table II is due to the fact that the experiments were of such scale that the effect of the weight of the material was small as compared with the transmitted vertical pressure. Furthermore, there appears to be no assurance that the principle of superposition on which the general equation for bearing capacity is based is applicable.

MR. W. S. HOUSEL[6] (*author's closure by letter*).—The discussions presented call attention to several points which appear to require elaboration. Both Mr. Krynine and Mr. Morrison discuss the question of statistical isotropy as applied to granular masses. An excellent discussion of isotropy is given by A. E. Cummings[7] wherein he accepts the definition of isotropy given by Planck which states in part that:

> "The necessary and sufficient condition for a body to be elastically isotropic—that is, that it should have no favored directions at all—is that its elastic constants should all be invariant with respect to any change of the coordinate system. . . ."

Mr. Cummings takes the position that this generally accepted definition of isotropy precludes the idea of different degrees of isotropy as described by Krynine[8] in his "statistical isotropy." In the light of the discussion of this subject in the writings of investigators of soil mechanics for the last year or two, the author is inclined to Mr. Cummings' view, namely, that a body is either isotropic or not and there can be no compromise in the application of the conception of isotropy. On the other hand it is the author's opinion that statistical regularity, described by Krynine as "statistical isotropy" is a very valuable and necessary conception if any rational method of expressing the behavior of soil masses is to be forthcoming.

Coulomb's internal friction theory in a sense does lead to a favored direction as it deduces failure on a plane of minimum resistance which does not coincide with the plane of maximum shearing stress. Mr. Morrison finds it hard to accept this statement and cannot understand the author's objection to classical methods. This point does not involve a question of viewpoint or a different way of describing the same thing. It is a mathematical necessity of static equilibrium, that in any case of internal stress there must be two principal stresses at right angles to each other and planes of maximum shear at 45 deg. with the principal planes. If the concepts of the classical internal friction theory are accepted, it is also a mathematical necessity that failure occur on planes making an angle with the principal planes equal to 45 deg. plus or minus one-half the angle of internal friction.

This inconsistency was pointed out in the paper and it is the author's opinion that the assumption of isotropy must be given up when dealing with materials which exhibit granular characteristics. It appears further that it is only a remote possibility that any aggregation of discrete particles will ever achieve a mechanical arrangement in which there are no favored directions as far as internal resistance is concerned.

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[6] Research Consultant, Michigan State Highway Dept.; Associate Professor of Civil Engineering, University of Michigan, Ann Arbor, Mich.

[7] A. E. Cummings, "Distribution of Stress Under a Foundation," Author's Closure, *Proceedings*, Am. Soc. Civil Engrs., Vol. 62, p. 720, May, 1936.

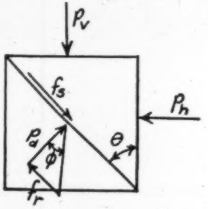
[8] Discussion by D. P. Krynine, "Distribution of Stresses Under a Foundation," *Proceedings*, Am. Soc. Civil Engrs., Vol. 61, October 1935, p. 1253.

The general concept of the elementary soil arch picturing the mutual support of adjacent particles recognizes the differences in internal resistance depending upon the orientation of an elementary arch with respect to the direction of applied force.

The author can see no objection to picturing the behavior of such a granular mass in terms of the behavior of individual particles of the elementary soil arch. Of course, as pointed out by Krynine, the angle of pressure transmission may vary through a wide range for individual particles, but given a sufficiently large number of individual particles the entire mass may be subjected to ordinary statistical analysis. In these terms such behavior may be represented "as if there were a hypothetical mass composed of grains of equal diameter and possessing a constant angle of distribution θ." This is nothing more than statistical regularity and may be applied to any phenomenon dealing with a wide range of probabilities, and does not imply isotropy in the accepted sense.

If it be generally accepted that granular masses are not isotropic the classical treatment of internal friction is not necessarily invalidated. The real weakness of the theory is that it fails to produce a criterion of failure. It was pointed out in the paper that a limiting ratio of the principal stresses cannot be set by tangential resistance on some inclined plane. In fact, the tangential resistance is controlled by the ratio of the principal stresses and is always equal and opposite to the tangential stress imposed. Thus failure or displacement of portions of the mass should never take place due to overcoming the tangential resistance on any plane.

This point may be best illustrated by the following demonstration:



Let θ = angle of transmission,
f_s = shearing stress on inclined plane,
f_r = frictional resistance on inclined plane, and
ϕ = angle of internal friction.

$$f_r = p_d \tan \phi \text{ (by definition)}$$

$$p_h = p_v \tan^2 \theta \tag{1}$$

$$p_v = \frac{p_d}{2 \sin^2 \theta} \tag{3}$$

$$f_s = \tfrac{1}{2}\, p_v\, (1 - \tan^2 \theta) \sin 2\,\theta \tag{4}$$

$$f_s = \frac{p_d}{4 \sin^2 \theta} (1 - \tan^2 \theta) \sin 2\,\theta$$

$$f_s = p_d \cot 2\,\theta$$

$$\theta = 45 - \phi/2 \text{ (by internal friction theory)}$$

$$f_s = p_d \cot (90 - \phi) = p_d \tan \phi$$

$$f_s = f_r$$

Referring to the element of mass shown above and considering that p_v is the applied pressure, it appears to the writer that one must be driven to the conclusion that failure or displacement of a portion of the mass can only be due to the application of a pressure which requires a larger horizontal thrust p_h than the adjacent element can supply. This is the basic consideration on which the "arch theory" of internal stability is formulated. Regardless of the nature of internal shearing resistance, whether it is friction, interlocking of particles, or something else, the author has been unable to escape this result. Of course the introduction of cohesion affects this consideration but internal stability was carefully defined to exclude cohesion.

In purely cohesive materials and in the total absence of granular characteristics, failure occurs when the difference between the principal stresses exceeds a given amount, or the shearing stress exceeds the shearing resistance on the plane of maximum shear. When cohesion is introduced into a granular structure it becomes more difficult to determine which influence is preponderant and it is perhaps impossible to define the plane of failure except by actual test. The bulk of experimental evidence available from compression test data appears to indicate that the structure or mechanical arrangement controls, as the angles of failure depart substantially from the angle of maximum shear.

There are several other points raised by Mr. Morrison involving differences in understanding of certain terms which the author feels deserve comment. The definition of internal stability given in the paper and on which the subsequent development is based does not conform to Mr. Morrison's definition. Stability is, of course, a relative term. A loose granular mass has less stability than a compact granular mass when stability is measured in terms of the applied force which may be sustained without displacement of portions of the mass. In other words, the compact granular masses investigated by the author have just as definite an ultimate load which they fail to support as do the loose granular masses, the only difference being in amount. So far as the author is aware, the measure of stability either of a structural member or of an arrangement of particles is generally accepted to be the ultimate load which may be supported without collapsing the system.

Mr. Morrison does not agree that friction is characteristic of a state of motion. He defines friction, "as that force which tends to oppose *motion* produced by other forces." This definition fails to dissociate motion and frictional force and if it did there would be no distinction between friction and any other type of internal resistance such as cohesion. As stated in the paper, Rankine based his development solely on the "friction of motion" and discounted static friction as being unreliable under practical conditions. As far as the author is concerned it still appears that friction necessarily depends on a state of motion and merges into other types of resistance or is lost altogether in a condition of static equilibrium.

In his discussion of the physical nature of friction Mr. Morrison overlooks the careful distinction between stability and cohesion laid down as the basis of the paper. The author included in cohesion all "forces of molecular origin, characteristic of microscopic and submicroscopic matter." Unless this distinction is recognized and cohesion includes all "atomic forces closely related to adhesion" acting on contaminated surfaces, or on any surfaces brought into molecular contact, much of the subsequent deduction becomes unsound. The same thing applies to the effect of moisture. It is intended that granular materials are of sufficient size that capillary forces are negligible and if not that they will contribute to cohesion in common with all other molecular forces.

Mr. Morrison points out that the data presented in the paper measure the angle of pressure transmission at the point of failure. He raises the question as to what this angle would be at loads less than the ultimate. The test conditions were so set up that a constant lateral pressure was maintained throughout the test while the applied vertical pressure was gradually increased until failure occurred. This was accomplished by locking the loading device against reverse movement so that the test specimen could not be disturbed by the applied lateral pressure. Under such conditions there was no opportunity to observe the relation between vertical and lateral pressure at any load except that which exceeded the ultimate stability of the system. It seems fair to conclude, however, that inasmuch as there were very small deformations at loads less than the ultimate, the mechanical arrangement of particles was practically the same as that which existed when the limiting value of stability was reached. If this deduction is valid and it is accepted that mechanical arrangement is the controlling factor, then the angle of pressure transmission θ is essentially constant until that arrangement is broken up.

Mr. Morrison raises a question regarding the two sources of horizontal thrust recognized in the development of the equation for bearing capacity and illustrated in Fig. 11 by the term Kwh. In this connection it may be pointed out that both the weight of the material and the vertical components of stress from the loaded area, acting on an element adjacent to the central column, are zero at the surface and increase with the depth for at least a limited depth. In the case of vertical components from the loaded area, the increase is not a linear function and at depths somewhat in excess of the diameter of the loaded area these forces decrease and become negligible as compared with the weight. However, the ultimate stability under vertical load is determined within a depth not greatly in excess of the diameter of the loaded area (see Fig. 10), and the approximation involved in assuming a linear variation in pressure transmitted appears to be satisfactory. If this were not so, the factor K evaluated from bearing capacity tests would not be so nearly constant as shown in Table II.

After all, the test of any theory is the accuracy with which it describes the results of observation. The author would take the position in this case that the superposition of two sources of resistance enables one to predict ultimate bearing capacity and to describe the dimensional effects involved which are entirely omitted by the conventional development of internal friction. While the submitted data are limited to a range of bearing area from 4 sq. in. to 32 sq. in. the extension to larger sizes has been successfully made in one case. The author is convinced that the principles involved are theoretically sound and that they are, in the broader aspects, substantiated by experimental results.

TESTING AND DESIGN OF STABILIZED SOIL MIXTURES

By C. A. Hogentogler, Jr.[1]

In a paper presented before the Society in 1934 (1)[2], Hogentogler and Willis stated that soil tests may be arranged in four groups depending on whether they are performed upon soil in (*a*) the dried and powdered state, (*b*) a specially compacted state, (*c*) the natural undisturbed state, or (*d*) a remolded state at natural moisture content.

Procedures for testing soils in the dried and powdered state were published by the Society as tentative methods in 1935 (2).

The research in soil mechanics at George Washington University under the Department of Civil Engineering headed by Prof. F. A. Hitchcock has for one of its principal objects the development of apparatus and test methods to disclose and measure the properties of soils of the second group—those in compacted or otherwise stabilized state. Close contact with the construction of soil road surfaces and dams by the U. S. Forest Service is affording a means for correlating the performance of compacted soils in service with their characteristics as determined in the laboratory.

According to the U. S. Bureau of Public Roads (3) stabilized soils may be of two kinds: (1) specially designed graded soil mixtures, supplemented possibly by admixtures of deliquescent or water retentive chemicals such as calcium chloride and common salt, compacted partly during construction and partly by traffic afterward to a high but qualitative degree of density; and (2) poorly graded and fine-grained soils with admixtures of electrolytes, fillers, or insoluble adhesive binders such as bituminous materials and portland cement, compacted during construction at optimum moisture or binder content to a quantitative degree of density, predetermined by test, and covered with a bituminous treatment or thin wearing course.

Tests for Graded Soil Mixtures

Well-graded materials should consist of (*a*) large and small aggregate proportioned to furnish the required resistance to abrasion and provide the interlocking which prevents sliding of soil grains; and (*b*) enough binder soil of the quality to provide the mixture with the capillarity required for stability in wet weather, and the cohesion or cementing strength required to maintain stability during dry weather, after capillary moisture has evaporated.

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[1] Research Associate, Department of Civil Engineering, George Washington University, Washington, D. C.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 482.

The abrasive resistance and the degree of interlocking of soil mixtures is indicated qualitatively by the grading of the granular fractions as disclosed by the mechanical analysis. Two tests of the first group, those for determining the liquid limit and the plastic limit of the dried and powdered fraction passing the No. 40 sieve, serve to distinguish between the moisture contents at which soil moisture acts as a lubricant and as a cohesive medium and indicate the quality of binder soils.

In colloidal suspensions, the soil particle may be considered to be encased in a film of adsorbed water or hydration and suspended in a body of free water as illustrated in Fig. 1. The free water has the freezing point, the surface tension and the viscosity of water in bulk. Adsorbed films, in contrast, have higher boiling points, lower freezing points, greater surface tension and are more glue-like than free water (4).

When a soil suspension is set aside, the soil particles will gradually settle to the bottom and form a sediment. The porosity, or moisture content, of this sediment at first is so high that the sediment has all the properties of a fluid, that is, it will deform due to its own weight to acquire the shape of any vessel in which it is placed. The minimum moisture content of the soil in this state is the true lower liquid limit (2) although the test result, the liquid limit as determined by the Atterberg tests, is somewhat less.

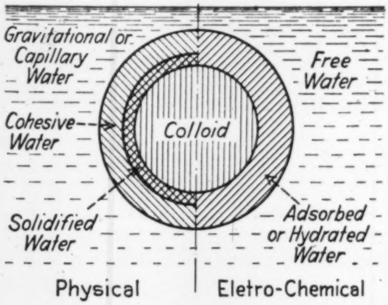


FIG. 1.—Illustrating Types of Soil Moisture.

When the moisture content is reduced below the lower liquid limit, the soils become stiff enough to require the application of external pressure to produce deformation. The required pressure gradually increases with continued evaporation until at what has been called the critical moisture content, the ratio of lubricating to cohesive moisture becomes so small that the cohesive water abruptly becomes the dominating influence on the performance of the soil. The resistance to deformation then increases considerably and abruptly. The critical moisture content has been found to equal 75 per cent of the liquid limit for non-plastic soils and the plastic limit for plastic soils (5).

TESTS FOR FINE-GRAINED SOILS

The density to which poorly graded or fine-grained soils should be compacted; the one moisture content, termed the "optimum moisture content," at which this density is produced by specific compactive effort; and the resulting stability are disclosed by tests devised by R. R. Proctor (6).

In the compaction test approximately 5 lb. of dry soil are mixed thoroughly with different amounts of water and compacted in a brass cylinder 4 in. in diameter and about $4\frac{1}{2}$ in. deep. mounted on a removable base plate and fitted with a detachable collar 2 in. high, to hold the loose soil in place while compacting. The soil is compacted in three layers, each layer receiving 25 blows from a $5\frac{1}{2}$-lb. cylindrical rammer having an end area of about 3 sq. in., and dropped from a height of 1 ft. above the soil. The soil is then struck off to the level of the cylinder, weighed, and the stability determined by observing the pressure required to penetrate the soil at the rate of $\frac{1}{2}$ in. per sec. with a plunger of known end area (see Fig. 2). A small sample of the compacted soil is oven-dried to determine the moisture content.

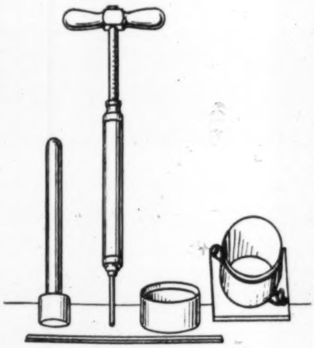


FIG. 2.—The Proctor Compaction Test Apparatus.

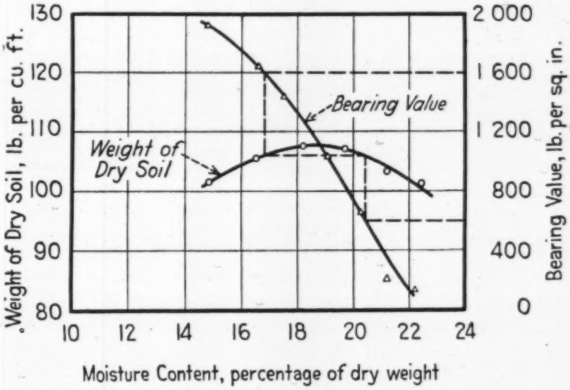


FIG. 3—Relations Furnished by the Proctor Tests.

The effect of moisture on the compacted densities of the representative samples is shown graphically by plotting the wet and dry densities of the compacted soil, expressed in pounds per cubic foot, against moisture content. The stability readings, expressed in pounds per square inch, are also plotted against moisture content to show the effect of moisture on stability.

Two curves resulting from the test, shown in Fig. 3, illustrate the significance of the compaction data. The dry weight-moisture content curve discloses that for this soil a moisture content of about 19 per cent is required, if maximum compaction is to be obtained. The corresponding stability is about 1100 lb. per sq. in. Increase or decrease of moisture from the optimum will ultimately result in lowered stability.

SCOPE OF PLASTICITY AND PROCTOR TESTS

The suitability of the plasticity tests for identifying dried and powdered soils passing the No. 40 sieve, especially those of the plastic varieties, has never been questioned. Much remains to be learned, however, regarding the significance of such test data for disclosing the performance of the entire soil mortar fraction passing the No. 10 sieve of both plastic and non-plastic or friable soils and in the compacted or stabilized state.

The Proctor tests have proven suitable for use in the field for purposes of control during the construction of earth fills. For laboratory purposes, however, the plasticity needle, as originally designed, is of limited value because of the possibility of prohibitive personal error. This is due to several causes: (*a*) it is almost impossible to obtain truly vertical movement during penetration; (*b*) the position in which the operator is forced to stand makes accurate measurement of the depth of penetration very difficult, especially when the soil bulges adjacent to the area penetrated; (*c*) a constant rate of penetration is very difficult to attain partly because of (*b*), but more because the movement of the handle, due to deflection of the force-indicating spring, can be several times the movement of the needle footing. In addition, the resistance to penetration, even at the optimum moisture content, is often found to be greater than the maximum of 2000 lb. per sq. in. which can be measured with the original needle.

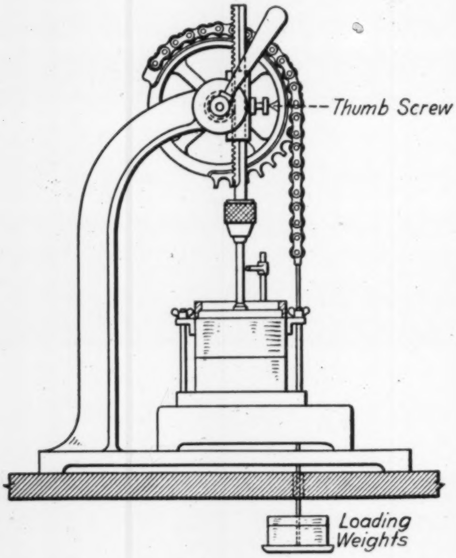
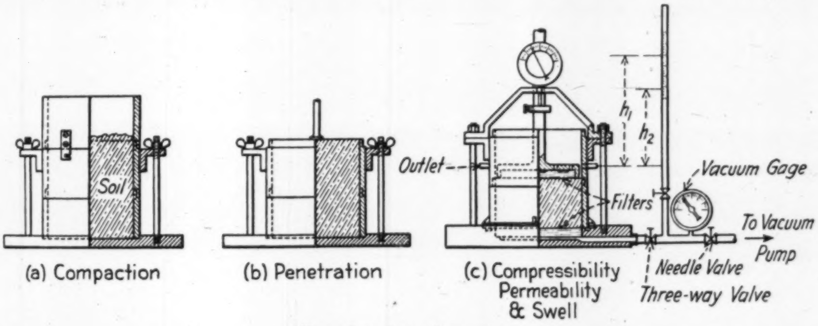


FIG. 4.—Penetrometer with Compaction Cylinder in Place.

An improved apparatus, therefore, was developed for obtaining information on the stability, capillarity and permeability of compacted soils. Provision was made in its design for supplementary attachments for obtaining information on the swell and compression characteristics of compacted soils.

The design shown in Fig. 4 is essentially that of a press which insures vertical movement of the plunger, accurate measurement of the depth and, hence, control of the rate of penetration, with provision for applying constant load. The pressures are determined by use of a platform balance of 300-lb. capacity and not subject to appreciable vertical movement. Depth of penetration is measured by a pointer referring to graduations on the needle stem. There are 7 penetration footings with end areas ranging from $\frac{1}{20}$ to 1 sq. in. Pressures up to 6000 lb. per sq. in. can be measured.

The supplementary parts, shown in Fig. 5, include: one compaction cylinder of 4 in. inside diameter and $6\frac{1}{2}$ in. high made in three segments, a bottom one 2 in. high, a middle one $2\frac{1}{2}$ in. high and a top one 2 in. high as shown in Fig. 5(*a*); a base plate $\frac{3}{4}$ in. thick and 6 in. square, shown also in Fig. 5(*a*); one filter base plate 6 in. square and 1 in. thick machined so as to receive a filter stone 4 in. in diameter and $\frac{1}{2}$ in. thick and to permit the free flow of water to and from the bottom of the filter stone as shown in Fig. 5(*c*). Attached to this filter base is a combination permeameter and capillometer which consists of a graduated glass standpipe $\frac{1}{2}$ in. in diameter, a vacuum gage, one three-way valve and one needle valve connected by tubing as shown. Also, as shown in Fig. 5(*c*) the upper segment of the cylinder, required when the apparatus is used as a permeameter, is provided with an outlet pipe. A further attachment is a plunger fitted with porous filter stone as shown in Fig. 5(*c*) and machined to fit neatly as shown.



(a) Compaction (b) Penetration (c) Compressibility Permeability & Swell

FIG. 5.—Attachments.

ESSENTIALS OF TEST PROCEDURE

The proposed complete testing procedure consists of the following steps:

(*a*) With the apparatus arranged as shown in Fig. 5(*a*), the sample is compacted in the manner previously described for the compaction test.

(*b*) The sample is struck off level with the top of the second segment as shown in Fig. 5(*b*).

(*c*) The weight of the compacted sample is determined.

(*d*) The plunger is forced into the sample, readings of the balance being taken at intervals of $\frac{1}{4}$ in. penetration to a maximum of 2 in.

(*e*) The upper segment is detached, the sample struck off level with the top of the lower segment, and a small portion of the material removed in levelling off the sample is used to determine the moisture content.

(*f*) The solid metal base plate is replaced by the filter base plate, the segment with outlet pipe is attached, the filter plunger fitted into position, the thumbscrew tightened and the permeability and capillarity attachments connected as in Fig. 5(*c*). The three-way valve at the bottom of the standpipe is opened and the standpipe kept filled until water escapes from the outlet pipe. To determine the permeability, the time required for the water level to drop a given distance in the standpipe is then observed.

TABLE I.—GRADING AND PHYSICAL CONSTANTS OF SOILS INVESTIGATED.[a]

Soil	Mechanical Analysis							Physical Characteristics of Material Passing No. 40 Sieve					
		Particles Smaller Than 2 mm., per cent by weight								Shrinkage		Moisture Equivalent	
	Particles Larger Than 2.0 mm.	Coarse Sand 2.0 to 0.25 mm.	Fine Sand 0.25 to 0.05 mm.	Silt 0.05 to 0.005 mm.	Clay Smaller Than 0.005 mm.	Colloids Smaller Than 0.001 mm.	Passing No. 40 Sieve	Liquid Limit	Plasticity Index	Limit	Ratio	Centrifuge	Field
No. 1.	0	17	27	12	44	33	94	55	30	24	1.6	32	36
No. 2.	0	. .	. .	Flocculated	. .	. .	. .	32	9	19	1.8	31	25
No. 3.	0	. .	. .	Flocculated	. .	. .	. .	40	0	48	1.1	20	45
No. 4.	0	9	0	0	0	0	82	21	0	29	1.5	3	27

[a] Data furnished by the U. S. Bureau of Public Roads.

(*g*) The force produced by the tendency of the soil to swell may be read on the scale. The thumbscrew holding the plunger in position is now loosened and the amount of swell of the sample is observed.

(*h*) The compressive and expansive properties of the sample may be determined by observing the deformation-load and the deformation-time relationships under conditions of both increasing and decreasing load applications.

(*i*) At the conclusion of the loading test, if made, or otherwise at the conclusion of the permeability test, the maximum capillary rise is determined in a manner based upon a method originally suggested by J. H. Englehardt (7), a modification of which was described by Hogentogler and Willis (8).

The three-way valve is adjusted so as to close off the standpipe and provide access of water into the soil by connecting to a reservoir. By means of the needle valve, the vacuum is increased by increments until abrupt loss of the vacuum thus built up indicates that the moisture films in the sample have been broken.

Calculations are made and the results recorded as follows: The density of the wet sample in pounds per cubic foot is determined by dividing its weight in pounds by its volume ($\frac{1}{30}$ cu. ft.). The corresponding dry density of the sample in pounds per cubic foot is determined by the formula:

$$\text{Dry density} = \frac{\text{wet density}}{100 + \text{moisture content}}\ 100$$

The dry weight – moisture content, and stability – moisture content relationships are recorded graphically.

The permeability is determined on the basis of the relationships expressed in the following formula from Gilboy (9):

$$k = \frac{2.3\ a\ d}{A\ t} \log \frac{h_1}{h_2}$$

where k = coefficient of permeability in feet per second,
a = cross-sectional area of standpipe in square inches,
d = thickness of soil sample in inches,
A = cross-sectional area of soil sample in square inches,
h_1 and h_2 = initial and final vertical heights, respectively, of water level in standpipe above outlet pipe in inches, and
t = time in seconds required for drop from h_1 to h_2.

For the dimensions of the apparatus herein described the foregoing formula becomes:

$$k = \frac{1}{167\ t} \log \frac{h_1}{h_2}$$

The maximum capillary height in feet is given by multiplying the final reading of the vacuum gage (in pounds per square inch) by 2.3.

Information to be obtained by use of the apparatus as designed is indicated by the following experimental data which were furnished by use of a modified apparatus in use at the George Washington University. The cylinder containing the compacted soil samples was placed on a platform scale which deflected at the rate of $\frac{1}{32}$ in. per 75-lb. load. A drill press was used to apply the load.

Observations were made principally to obtain some light on how the stability of compacted samples is affected by such variables as (1) moisture content, (2) size of bearing area, (3) depth of penetration, (4) rate of penetration, (5) temperature, and (6) clay content.

Effect of Moisture Content:

The physical character and grading of four of the soils investigated are disclosed by the data given in Table I. Soil No. 1 represents a highly plastic clay; No. 2, a feebly plastic fine-grained soil; No. 3, a non-plastic pumice with relatively high capillarity; and No. 4, a cohesionless beach sand with neither plasticity nor detrimental capillarity. The samples were penetrated at a rate of approximately 1 in. per min.

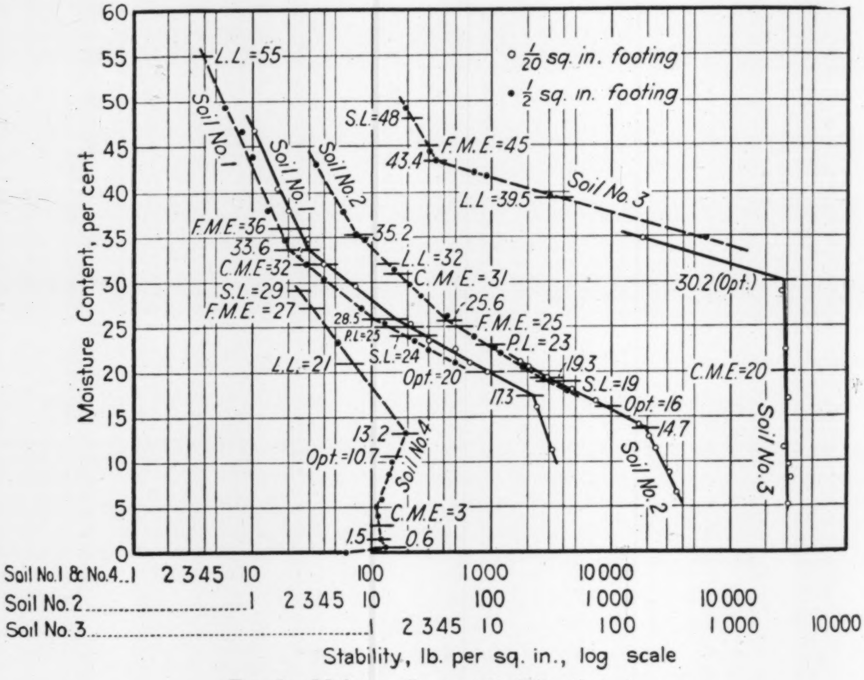


FIG. 6.—Moisture Content-Stability Relations.

L. L. = Liquid limit. S. L. = Shrinkage limit. P. L. = Plastic limit. C. M. E. = Centrifuge moisture equivalent. F. M. E. = Field moisture equivalent. Opt. = Optimum moisture content.

Data shown graphically in Fig. 6 reveal the relationships of stability to moisture content. They consist of a series of logarithmic curves each of which is similar in form to that of the flow curves obtained with the liquid limit device and described in the paper by Hogentogler and Willis (1). They are expressed by the equation:

$$W = A - B \log P$$

where W = moisture content in per cent by weight of dry soil,
A = moisture content at pressure of 1 lb., and
B = difference in moisture content corresponding to a difference of unity in the logarithm of the load P.

The curves for soils Nos. 1 and 2, the plastic soils, have three breaks. The significance of the breaks at the two higher moisture contents is not known at this time. The breaks in the same curves at the lower moisture contents, 17.3 per cent for No. 1 and 14.7 per cent for No. 2 undoubtedly represent the critical moisture contents. They are considerably below the plastic limits, as well as all the other routine subgrade soil test constants. Also they are slightly below the optimum moisture contents, thus indicating that more than the 25 blows of the tamper used in compacting the samples are required to reduce the optimum moisture contents to the critical moisture contents of the samples.

The curve furnished by tests on soil No. 3, the cohesionless pumice, has only two breaks, of which the one occurring at 30.2 per cent is the critical moisture content. It is approximately equal to 75 per cent of the liquid limit, which agrees with findings reported previously by the Bureau of Public Roads (5). For this material the optimum moisture content is equal to the critical moisture content.

As would be expected, a slight amount of moisture, (0.6 per cent) added to the dry sand (soil No. 4) causes an abrupt increase in stability. With increase in moisture content from 0.6 to 5 per cent bulking occurred with decrease in the density of the sample and, as shown by the curve for soil No. 4, a slight reduction in stability. With further increase in moisture content bulking ceased, the density of the compacted sand increased and the stability became greater until a moisture content of 13.2 per cent, the maximum which the sand would retain under conditions of free drainage, was reached. The stability of the sample when completely immersed is shown by the uppermost point on the curve at a moisture content of 23.3 per cent. Although shown by a straight line, the relationship of stability to moisture content between 13.2 and 23.3 per cent moisture is not known.

Effect of Size of Bearing Area:

According to the data shown in Fig. 6, stabilities observed for the highly plastic soil, No. 1, above the critical moisture content were smaller when the plunger of $\frac{1}{2}$ sq. in. end area was used than when the plunger of the smaller end area of $\frac{1}{20}$ sq. in. was used. For the feebly plastic soil, No. 2, the stabilities as indicated by the $\frac{1}{2}$-in. plunger also were smaller than those indicated by the $\frac{1}{20}$-in. plunger but only slightly so. For non-plastic soil, No. 3, the reverse was true, the larger stabilities being indicated by the plunger with the larger bearing area.

Only factor A in the equation $W = A - B \log P$ seems to have been influenced by the end area of the penetrating needle. Factor B, which is representative of the slope of the curve on a semi-log plot and also the moisture contents at which the breaks in the relationships occur, including the one indicative of the critical moisture content, seems to be unaffected by the size of plunger used.

Data obtained but not shown on Fig. 6 indicate no relationship between stability and area of plunger for soils compacted below the critical moisture content.

Effect of Depth of Penetration:

Data on the effect of depth of penetration upon observed stabilities are shown in Fig. 7. The absence of uniformity of stability at different depths of the compacted samples is apparent. In the plastic soils, Nos. 1 and 2, the tendency for the pressure to build up with increase in the depth of penetration is considerably less than in the cohesionless pumice and beach sand, soils Nos. 3 and 4.

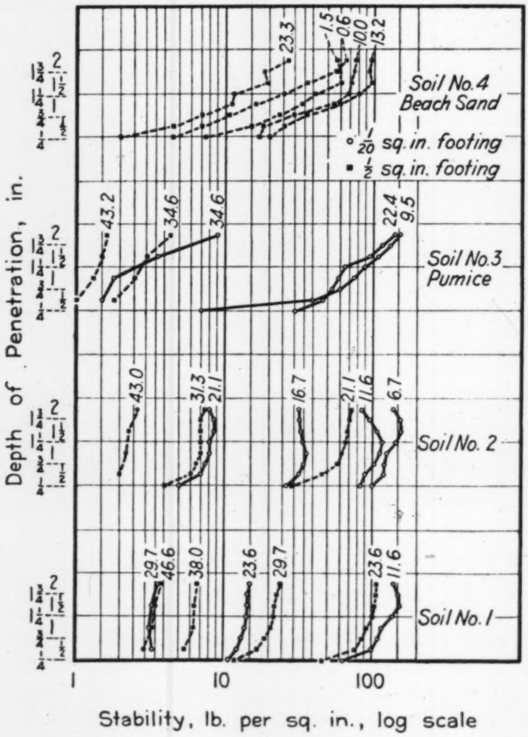


FIG. 7.—Stability-Penetration Relations.

The numbers at the top of each curve represent the moisture content in per cent by weight.

In order to determine the effect of the variation of stability with the depth of penetration, as indicated by the curves, Fig. 7, the stabilities observed at different depths were averaged, first for depths of $\frac{1}{4}$ to 2 in., and second from 1 to 2 in., inclusive. In comparison with the maximum readings obtained throughout the range of $\frac{1}{4}$ to 2 in. depths, it was found that while the stabilities in the three instances vary somewhat for the different soils, the slopes of the curves are practically the same and the indicated breaks occur at approximately the same moisture contents. Therefore, the maximum values obtained in a given depth of penetration seem suited for use in indicating the stability.

The non-uniformity of density just discussed does not necessarily decrease the accuracy of determinations of density and optimum moisture contents.

Effect of Rate of Penetration:

A limited number of observations for determining the effect of rate of penetration indicated that within the range of 0.6 in. per min. to 2.4 in. per min. there was no change in the indicated stabilities.

Effect of Temperature:

Data just published by the Bureau of Public Roads (3) discloses that for equal moisture contents the lower the temperature of the soil (above the freezing point) the higher will be its stability. This is because at lower temperatures the glue-like water films are thicker than at higher temperatures. Because of this increased thickness it has been found by Baver and Winterkorn (10) that the hydrated, or glue-like water, per gram of soil colloid may be as much as doubled by temperature change from 99 to 30 F. As a result, sedimentation proceeds at a slower rate, more effort of compaction is required to produce equal density and the flow of water through soils occurs at a slower rate in cold than in warm weather.

With a plunger of $\frac{1}{20}$ in. end area in the penetrometer, the maximum stability over the range of $\frac{1}{4}$ to 2 in. depth of penetration was observed to be 1660 lb. per sq. in. at 100 F. and 2000 lb. per sq. in. when the temperature of a compacted sample of soil No. 1 was reduced to 54 F. With a plunger $\frac{1}{10}$ in. in area, there was observed an increase in the stability of a sample of soil No. 2 from 180 lb. at 93 F. to 223 lb. when the temperature was lowered to 59 F. It has been found that because of the effect of temperature on the thickness of hydrated films on colloids and on the viscosity of water, soil test results indicative of specific consistencies of sample (such as liquid limit and plastic limit), are influenced by temperature change. For the same reason, it can be expected that constants indicative of compressibility, capillarity, and permeability are likewise affected by temperature change.

Effect of Clay Content:

The drier the conditions under which graded road mixtures are to serve, the higher should be their clay contents. Thus, road surfaces exposed to the sun should, as a rule, have higher percentages of clay than mixtures which are to serve as base courses protected by impervious surfacing from evaporation.

In comparison to the information available on the design of road surfaces there exists but little to guide in the design of base courses.

In order to determine the clay content for maximum stability under very wet conditions, graded granular material and clay binder available on particular jobs were combined in varying proportions and then placed in molds such as the Proctor compaction cylinder at moisture contents high enough to permit the mixture to slump to maximum density. The samples were then drained of free water and tested with the penetrometer. Figure 8 shows the type of data obtained. It will be noted that with only capillary moisture in the soil there is an optimum clay content, about 3.5 per cent (8 per cent based on material passing No. 40 sieve) in this case, at which the mixtures have maximum stability.

The combinations of any kinds of soil materials required to provide maximum stability at any moisture content or any degree of compaction can be determined by using the same testing procedure but varying the method of preparing the sample. Instead of testing at maximum capillary moisture, as noted above for base course soil, samples of materials to be used for road surfacing may be tested at lower moisture contents. Data obtained to date indicate that the optimum clay content of mixtures increases as the moisture content at which the samples are compacted decreases. This indicates the validity of the requirement which calls for variation of clay content with change in conditions of moisture under which the roads are to serve.

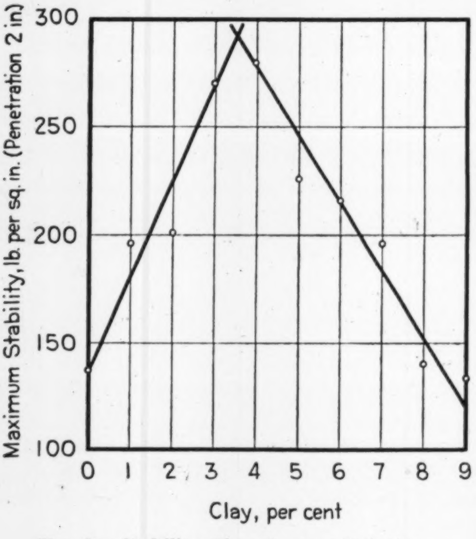


FIG. 8.—Stability-Clay Content Relation.

SUMMARY

The work in its present status leaves much to be learned. However, the following may be included among definite indications.

1. The moisture content – stability relationships for the maximum and the average stabilities indicate that the effect of lack of uniformity in compaction may be largely overcome if a sufficient number of observations are made and to a depth greater than the thickness of each compacted layer of the sample. Nevertheless, in research on basic soil physics, it seems desirable that a method of compaction be used which will produce more uniform density throughout the thickness of the sample than the method originally proposed by Proctor for use in field control test.

2. Observations should be made as nearly as possible at constant temperature.

3. The plastic limit as furnished by the Atterberg test obviously does not disclose the true plastic limit or critical moisture content of soils compacted in the manner used in the Proctor tests.

4. If observed stabilities are indicative of shear strength, then the assumptions that at the liquid limit all soils have equal shear strength and that for plastic soils between the liquid limits and plastic limits a constant logarithmic relationship between moisture content and shear strength for each soil exists, do not seem to hold true for compacted soils.

The more the stability depends upon internal friction, instead of cohesion, the greater seems to be the shear strength as indicated by the penetrometer at the liquid limit of soils. The stability of soil No. 1 (see Fig. 6) at the liquid limit is 4 lb. per sq. in., soil No. 2, 14 lb., Soil No. 3, 36 lb., and for soil No. 4 it is not likely to be less than 52 lb., the stability under complete immersion. This indicates that the relative effect of shocks to produce flow in the liquid-limit test differs from the resistance to penetration in the stability tests depending upon the relative cohesion and internal friction of the soil.

5. Because of the effort required to make shear tests according to currently used procedures and the controversial aspect of proposed procedures for applying the results of such tests in practice, it would seem highly desirable to investigate the possibilities of using the simple penetration test just described as a substitute. With change of end area of the plunger, only the factor A of the moisture content – stability curves changes, being smaller and decreasing with increase in the size of the footing as the plasticity of the soil increases and being larger in non-plastic soils. Because of this, it is quite likely that by means of the stabilities determined with plungers of different end areas the relative amounts of cohesion and internal friction which furnish the total stability could be estimated.

6. The data indicate: (*a*) Soils compacted to maximum density at low temperature may soften due solely to the water liberated from the adsorbed films by a rise in temperature; (*b*) for equal compaction, greater densities may be attained at higher temperatures, and to obtain similar

densities more work of compaction will be required at the lower temperatures; (*c*) the outcome may be very unsatisfactory when clay structures are compacted to a given extent without reference to the type or extent of compaction.

7. The penetration tests seem to provide a means for designing graded mixtures on a quantitative basis, with stability as a criterion, rather than the qualitative tests for grading and plasticity. If the samples are prepared to represent as closely as possible the state of the mixtures in service, it is very probable that limiting stability values can be set up as a basis of satisfactory performance.

8. Finally, there seems to be no consistent relation between any of the routine subgrade test constants and the moisture contents at which abrupt changes in the stability of compacted soils occur, with the possible exception that the critical moisture contents of non-plastic soils equal 75 per cent of their liquid limits. It should be emphasized that this does not affect the value of the routine tests for serving the purpose for which they are used, that of identifying dried and powdered subgrade soils according to types, and fine-grained soils for use as binder in graded soil mixtures. For the latter purpose the liquid and plastic limit tests, especially, have been proved by experience to be suitable and convenient tools.

For complete investigation of the properties of soils in compacted or other stabilized state, however, a new set of constants equal to the moisture contents at which abrupt changes in stability occur, as disclosed by the penetrometer, seem well worth investigating.

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DISCUSSION

Mr. D. M. Burmister.[1]—I wish to emphasize the fact that we must now think of soil as an engineering material. The use of soil for foundations, earth structures, highways, etc. must rest on the knowledge of the true physical nature of soils, and on the known behavior of soils under all possible conditions. Our knowledge has been materially increased by important soil investigations, and through the development of soil testing methods, which make it possible to determine certain essential physical properties of the soil. This knowledge of the character and behavior of soil has reached a state where it can be said that there are certain tangible facts about soil which can be used for engineering purposes in much the same way that the results of physical tests on the common structural materials, such as steel and concrete, can be used for design purposes.

For example, one of the most significant facts that has appeared recently is the important effect of moisture content upon the density to which a given soil may be compacted by means of a given method. The basic principles of soil compaction were first presented by R. R. Proctor, who applied them to the compaction of soils in the construction of rolled-earth dams. The following facts stand out:

1. There exists a definite but narrow range of moisture content that gives the maximum density to which a soil may be compacted by a given compacting force.
2. The moisture content and the density are characteristic for a given soil and depend on certain essential physical properties of the soil.
3. This leads to the concept of an optimum condition of moisture content and density as soil constants for a given soil.

However, it is at present more important to determine the combined influence of all of these soil properties than to be able to define the exact nature of the specific characteristics themselves, or the dependence of behavior of soil upon each.

There are, therefore, available two soil constants which can be used for describing soils more adequately and for defining a little more closely their engineering properties, so as to predict their behavior as highway subgrades or as fill material in dams. Both are subject to wide variation in moisture content. It is essential to know something of the extent of softening that may take place as a result of saturation.

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[1] Research Laboratories, Columbia University, New York City.

It is believed that the basic principles of soil compaction have an even more significant and universal application. If a definite density is attained by a given soil at a given moisture content under a certain compacting force, then, by the *Reciprocal Law*, the load that the given soil, in the natural state and in the same condition of density and moisture content, is capable of supporting without appreciable settlement should bear some definite relation to the above compacting force.

Recent studies at Columbia University have shown that very definite relations exist between the basic principles of soil compaction and the grading of the soil. But it is necessary to revise some of our conceptions of the significance of the grading of the soil and its interrelationships with other soil properties.

Future progress in the science of soils for engineering uses must be along lines (1) of close and accurate observation of soil phenomena and soil behavior under actual conditions encountered in practice; (2) of the correct interpretation of the observed facts; and (3) of their correlation with the results of physical tests.

The important thing which should be emphasized, however, is that soil investigations are yielding soil constants by which soils can be adequately described for engineering purposes.

THE EFFECT OF MINERAL FILLERS ON THE SERVICEABILITY OF COATING ASPHALTS[1]

By O. G. Strieter[2]

Synopsis

The durabilities of filled and unfilled coating asphalts are compared, both in outdoor and in accelerated exposures. The tests show that in general the durability to weathering of coating asphalt can be improved by the addition of mineral fillers and that there is a difference in the effectiveness of various fillers. The data demonstrate the similarity between outdoor and accelerated exposures.

Introduction

Asphalt shingles and roll-roofing are made by impregnating felt with a relatively soft asphalt and then surfacing with an asphalt that is considerably harder, the latter being known as a coating asphalt. In recent years the practice has become general to mix finely ground slate, limestone, and similar mineral fillers with the asphalt to obtain a coating which is less affected by sunlight and less subject to plastic flow.

The effectiveness of fillers can be compared on the basis of equal volumes, or equal surface areas, or equal weights of fillers. In the present investigation the effects of fillers on coating asphalt were compared only on the basis of equal weights, since this is the simplest and quickest way to compare filled and unfilled coating asphalts. Accordingly, a variety of asphalt-filler mixtures were prepared on a weight basis and tested. Specific gravities, compacting weights, sieve analyses of the mineral fillers, and softening points and penetrations on the asphalt-filler mixtures were determined. The weather-resisting properties of the asphalt-filler mixtures were determined by exposing the mixtures outdoors in various localities and to the accelerated weathering cycle. From the data so obtained comparisons were made between the filled and unfilled coating asphalts, and between the effects of accelerated weathering and outdoor exposures.

Preparation, Compositions, and Mode of Exposure of Panels

For preparing the asphalt-filler mixtures two asphalts of different softening points were used. Both asphalts were made from the same crude

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associate for the Asphalt Shingle and Roofing Institute at the National Bureau of Standards, Washington, D. C.

TABLE I.—COMPOSITIONS, PHYSICAL PROPERTIES, AND DURABILITIES OF ASPHALT-FILLER MIXTURES.

Specimen	Asphalt-Filler Mixture, per cent			Percentage of Hard Asphalt in Total Asphalt	Kind of Filler	Penetrations in 0.01 cm. units[a]			Durability	
	Hard Asphalt	Soft Asphalt	Filler			At 32 F., 200 g., 60 sec.	At 77 F., 100 g., 5 sec.	At 115 F., 50 g., 5 sec.	1½ yr. Outdoor Exposure, Group Classification[b]	Life in Accelerated Test, cycles
No. 1...	47	18	35	72	Green slate flour, filler No. 1	6	9	13	No. I	62
No. 2...	62	13	25	83		6	9	16	No. II	45
No. 3...	73	12	15	86		7	9	17	No. II	37
No. 4...	47	18	35	72	Dolomite, filler No. 2	6	10	18	No. II	37
No. 5...	60	15	25	80		6	10	17	No. II	28
No. 6...	73	12	15	86		7	10	17	No. III	28
No. 7...	47	18	35	72	Dolomite, filler No. 3	6	9	15	No. II	40
No. 8...	60	15	25	80		8	11	18	No. II	3
No. 9...	73	12	15	86		7	10	17	No. III	28
No. 10...	47	18	35	72	Limestone, filler No. 4	6	9	16	No. II	43
No. 11...	60	15	25	80		6	10	17	No. II	37
No. 12...	73	12	15	86		6	9	18	No. III	33
No. 13...	47	18	35	72	Peach-bottom slate, filler No. 5	6	8	15	No. I	65
No. 14...	60	15	25	80		7	10	16	No. I	62
No. 15...	73	12	15	86		7	8	18	No. II	43
No. 16...	47	18	35	72	Silica sand, filler No. 6	8	12	18	No. II	28
No. 17...	68	7	25	90		6	11	15	No. II	28
No. 18...	80	5	15	94		8	11	14	No. III	28
No. 19...	47	18	35	72	Greenstone, filler No. 7	7	11	14	No. I	65
No. 20...	60	15	25	80		7	10	16	No. II	46
No. 21...	73	12	15	86		7	10	17	No. II	40
No. 22...	—	65	35	0	Hydrated lime, filler No. 8	6	8	14	No. III	28
No. 23...	—	75	25	0		8	11	21	No. III	28
No. 24...	37	48	15	56		7	11	21	No. III	28
No. 25...	36	29	35	55	Trap rock, filler No. 9	6	9	15	No. II	43
No. 26...	53	22	25	70		7	9	17	No. II	37
No. 27...	72	13	15	85		7	9	16	No. III	28
No. 28...	13	72	15	15	Supercel, filler No. 10	8	10	22	No. II	40
No. 29...	41	44	15	48	Mica, filler No. 11	7	10	17	No. I	65+
No. 30...	23	42	35	36	Foliated talc, filler No. 12	6	10	16	No. I	65
No. 31...	44	31	25	58		7	9	18	No. I	65
No. 32...	66	19	15	78		7	9	18	No. II	48
No. 33...	49	16	35	75	Silica dust, filler No. 13	7	8	16	No. II	35
No. 34...	62	13	25	83		7	8	16	No. II	33
No. 35...	78	7	15	92		7	9	17	No. III	28
No. 36...	40	25	35	62	Silica dust, filler No. 14	7	9	18	No. II	35
No. 37...	54	21	25	72		6	9	16	No. II	30
No. 38...	72	13	15	85		6	9	16	No. III	28
No. 39...	49	16	35	75	Silica dust, filler No. 15	7	9	17	No. II	35
No. 40...	64	11	25	85		7	10	17	No. II	35
No. 41...	77	8	15	91		7	9	17	No. III	28
No. 42...	33	32	35	51	Slate flour, filler No. 16	7	10	17	No. I	65
No. 43...	50	25	25	67		7	10	17	No. II	51
No. 44...	68	17	15	80		7	10	17	No. II	43
No. 45...	44	21	35	68	Slate flour, filler No. 17	7	10	14	No. I	65
No. 46...	58	17	25	78		7	10	15	No. I	65
No. 47...	75	10	15	88		7	10	18	No. II	43
No. 48...	43	22	35	66	Limestone, filler No. 18	7	10	18	No. II	45
No. 49...	56	19	25	75		7	10	18	No. III	33
No. 50...	75	10	15	88		7	10	19	No. III	28
No. 51...	49	16	35	75	Limestone, filler No. 19	7	10	18	No. II	58
No. 52...	64	11	25	85		8	10	17	No. II	48
No. 53...	81	4	15	95		6	10	19	No. III	28
No. 54...	100	—	—	100		7	10	17	No. III	28
No. 55...	75	25	—	75		9	11	17	No. III	28
No. 56...	50	50	—	50		8	12	21	No. III	28
No. 57...	25	75	—	25		9	15	27	No. III	28

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[a] The results reported are the average of 3 or 4 determinations.

[b] Group No. I.—Outdoor panels showing no cracks to the eye or with magnifying glass, or, at most, some of the panels of a set showing faint cracks to the eye or with a magnifying glass. Group No. II.—Outdoor panels showing cracks to the eye. Group No. III.—Outdoor panels showing wide cracks to the eye.

petroleum in a single operation, differing only in the duration of the blowing. The softening points of the two asphalts were 194 and 225 F., respectively.

The "soft" and "hard" asphalts were so blended that, when combined with 15, 25 and 35 per cent by weight of various mineral fillers, the softening point of the resulting mixtures was approximately the same as that of the original "hard" asphalt, namely 225 F.

Table I shows the compositions and penetrations[3] of the asphalt-filler mixtures.

TABLE II.—PHYSICAL PROPERTIES OF THE FILLERS.

Filler	Kind of Filler	Particle Size of Filler			Oil-Absorption Ratio	Specific Gravity	Compacting Weight	Fineness Factor, *f*
		Passing No. 200 Sieve, per cent	Passing No. 100 but Retained on No. 200 Sieve, per cent	Retained on No. 100 Sieve, per cent				
No. 1....	Slate flour—Air floated No. 000 grade	81	17	1	0.7	2.86	1.35	1.1
No. 2....	Dolomite	92	7	1	0.5	2.85	1.63	0.8
No. 3....	Dolomite	85	14	1	0.4	2.88	1.73	0.7
No. 4 ...	Limestone	91	8	1	0.4	2.76	1.62	0.7
No. 5....	Peach-bottom slate—No. 000 grade	79	16	5	0.8	2.98	1.40	1.1
No. 6....	Silica sand	2	13	85	0.2[a]	2.69	1.74	0.6
No. 7....	Greenstone	65	24	12	0.6	3.08	1.66	0.9
No. 8....	Hydrated lime	99	2	—	1.3	2.39	0.83	1.9
No. 9....	Trap rock	97	3	—	0.7	2.94	1.52	0.9
No. 10....	Celite (Hy Flo Supercel)	100	—	—	2.9	2.52	0.30	7.4
No. 11....	Mica	42	28	30	2.2[b]	3.20	0.51	5.3
No. 12....	Foliated talc	100	trace retained on No. 200 sieve	—	1.1[b]	2.97	1.05	1.8
No. 13....	Silica dust	67	26	7	0.5[b]	2.69	1.68	0.6
No. 14....	Silica dust (prepared from filler No. 13)	100	—	—	0.5	2.69	1.63	0.7
No. 15....	Silica dust (prepared from filler No. 13)	—	100	—	0.4	2.69	[c]	—
No. 16....	Slate flour (prepared from filler No. 5)	100	—	—	0.9	2.98	1.30	1.3
No. 17....	Slate flour (prepared from filler No. 5)	—	100	—	0.7	2.98	[c]	—
No. 18....	Limestone (prepared from filler No. 4)	100	—	—	0.5	2.76	1.63	0.7
No. 19....	Limestone (prepared from filler No. 4)	—	100	—	0.5	2.76	[c]	—

[a] Does not possess plastic properties, but merely wets.
[b] Not a sharp end-point.
[c] Insufficient material for test.

In all, 57 different combinations of asphalts and asphalt-filler mixtures, including nearly all the fillers in commercial use, were prepared and surfaced on aluminum sheet metal (3 by 6 in.) to a thickness of 0.025 in.[4] Seven sets of such panels were prepared in duplicate, making 114 panels for each set. Six sets were exposed outdoors at Buffalo, N. Y., Manville, N. J., Chicago, Ill., Los Angeles, Calif., New Orleans, La., and Washington, D. C. The panels in the outdoor exposures were placed on racks at an angle of 45 deg. facing south. A seventh set of panels was subjected to accelerated weathering consisting of exposing the panels alternately to light from an inclosed carbon arc, water spray, and sudden temperature changes.[5]

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[3] Method of test is fully described in the Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1933 Book of A.S.T.M. Standards, Part II, p. 971.

[4] The method of preparing the panels is fully described in a paper by O. G. Strieter on "Accelerated Tests of Asphalts," National Bureau of Standards *Journal of Research*, Vol. 5, No. 1, p. 247 (1930); also in the Proposed Method for Accelerated Weathering Tests on Bituminous Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 381 (1933).

PHYSICAL PROPERTIES OF THE FILLERS

The effect of fillers in admixture with asphalt must depend not only upon the nature of the filler and its proportion in the mixture, but also upon certain physical properties such as the fineness and the tendency to absorb oil. For this reason some of the physical properties of the fillers were determined and are listed in Table II. As shown in Table II, fillers Nos. 14 to 19 were prepared from fillers Nos. 4, 5 and 13.

By oil absorption is meant the amount of oil which is required by a specific amount of mineral filler to form a paste. The method[6] employed in determining it consists in adding linseed oil gradually, drop by drop (by means of a small burette) to the filler, and after the addition of each drop thoroughly incorporating the oil with the filler by rubbing up with a sharp-edged steel spatula. The test is complete when exactly enough oil has been incorporated with the filler to produce a stiff, putty-like paste. The weight of the filler used divided by the density of the filler gives the volume of the filler. The number of milliliters of linseed oil required to form the paste divided by the volume of filler gives the number of milliliters of linseed oil absorbed by 1 ml. of filler, or the oil-absorption ratio.

The specific gravities of the fillers were determined by means of a pycnometer, using kerosine as the liquid. The details of the method have been described elsewhere.[7]

The compacting weights were determined by compacting the filler in a 100-ml. graduated cylinder by tapping the bottom of the cylinder against a padded table top. Enough filler was added at a time to the cylinder so that when compacted the volume would be about 10 ml. The tapping was continued for each 10 ml. until there was a dull sound which indicated that the filler was compact. This process was continued until 100 ml. of compacted volume was obtained. The weight of the filler per milliliter is called the compacting weight.

Since the compacting weights of fillers with different specific gravities cannot be directly compared, the compacting weights were used to calculate a "fineness factor," f, according to the following formula:[8]

$$f = \frac{\text{density} - \text{compacting weight}}{\text{compacting weight}}$$

The finer a given filler has been ground, the larger is its fineness factor.

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[5] The accelerated weathering test is fully described in the Proposed Method for Accelerated Weathering Tests on Bituminous Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 381 (1933).

[6] Standard Method of Test for Oil Absorption of Pigments (D 281–31), 1933 Book of A.S.T.M. Standards, Part II, p. 561.

[7] Standard Methods of Test for Specific Gravity of Pigments (D 153–27), 1933 Book of A.S.T.M. Standards, Part II, p. 568.

[8] Franz Pöpel, "Der Moderne Asphaltstrassenbau," Strassenbau-Verlag Martin Boerner, Halle, Germany (1929).

OUTDOOR EXPOSURES AND ACCELERATED TESTS

Behavior Toward Outdoor Exposure:

Asphalts exposed outdoors weather characteristically, depending upon their composition and upon the weather peculiar to the locality of exposure. Thus there is a marked difference in appearance in the six series of panels exposed in the various localities. The outdoor panels were examined after $1\frac{1}{2}$ yr. of exposure as described under the following headings:

Surface Oxidation.—Asphalts exposed to the weather form a film of oxidized material on their surface, which may be readily rubbed off. The amount of such oxidized material formed depends upon the nature of the asphalt, the intensity of the sunlight, and the amount of moisture.

As regards locality, the coatings exposed at New Orleans (NO) show the greatest surface oxidation, the Washington (W) coatings are next, followed by the Buffalo (B) exposures. The Manville (M) and Chicago (C) coatings show less surface oxidation than the NO, W and B coatings. The C coatings had been scrubbed before their return for inspection and so were difficult to classify. However, they resembled the M coatings as regards oxidation and surface configuration. When originally received, the Los Angeles (LA) coatings were covered with a heavy film of clay or dust. This film was of sufficient thickness to hinder the progress of weathering and this, together with the milder weather, resulted in coatings that showed no marked deterioration after $1\frac{1}{2}$ yr. outdoor exposure.

Cracking.—Aside from surface oxidation, asphalts crack upon continued exposure to the weather. This type of weathering is influenced by the composition of the coating and is also characteristic for each locality.

The B coatings cracked to the greatest degree but are closely followed by the M and C coatings. The NO and W coatings cracked less and the LA coatings, with a few exceptions, showed no cracks at all. As a rule the B, M and C coatings showed short cracks tending to form four-sided but irregular checks. The W coatings as a rule showed long, narrow cracks, whereas in the NO coatings the cracks were much wider.

Classification of Weathered Coatings:

The degree of cracking on outdoor exposure was made the basis for classifying the various coating mixtures. The panels were examined for cracks by visual inspection (unaided eye), and under the magnifying glass (× 12). Based on such an examination the outdoor exposures were classified in three groups (Table I) as follows:

No. I. Outdoor panels showing no cracks to the eye or with magnifying glass, or, at most, some of the panels of a set showing faint cracks to the eye or with a magnifying glass.

No. II. Outdoor panels showing cracks to the eye.

No. III. Outdoor panels showing wide cracks to the eye.

In the above classification the LA panels were not considered, since they showed relatively little change on weathering.

The rating of all the specimens was the same at each location of exposure with the exception of specimens Nos. 28, 32, and 43. These specimens were placed in group No. II on the basis of all exposures except at New Orleans. At New Orleans they showed no cracks and hence would have been placed in group No. I.

TABLE III.—ORDER OF DURABILITY OF ASPHALT-FILLER MIXTURES.

Specimen	Composition	Durability, number of cycles
GROUP NO. I		
No. 29....	15 per cent Mica Filler No. 11	65+
No. 45....	35 per cent Slate Filler No. 17	65
No. 30....	35 per cent Talc Filler No. 12	65
No. 31....	25 per cent Talc Filler No. 12	65
No. 13....	35 per cent Slate Filler No. 5	65
No. 42....	35 per cent Slate Filler No. 16	65
No. 46....	25 per cent Slate Filler No. 17	65
No. 1....	35 per cent Slate Filler No. 1	62
No. 19....	35 per cent Greenstone Filler No. 7	65
No. 14....	25 per cent Slate Filler No. 5	62
GROUP NO. III		
No. 6....	15 per cent Dolomite Filler No. 2	28
No. 9....	15 per cent Dolomite Filler No. 3	28
No. 12....	15 per cent Limestone Filler No. 4	33
No. 18....	15 per cent Sand Filler No. 6	28
No. 27....	15 per cent Trap rock Filler No. 9	28
No. 35....	15 per cent Silica Filler No. 13	28
No. 38....	15 per cent Silica Filler No. 14	28
No. 41....	15 per cent Silica Filler No. 15	28
No. 49....	25 per cent Limestone Filler No. 18	33
No. 53....	15 per cent Limestone Filler No. 19	28
No. 54....	Asphalt softening point 108 C.	28
No. 55....	Asphalt softening point 104 C.	28
No. 56....	Asphalt softening point 99.5 C.	28
No. 57....	Asphalt softening point 94 C.	28
No. 50....	15 per cent Limestone Filler No. 18	28
No. 22....	35 per cent Hydrated lime Filler No. 8	28
No. 23....	25 per cent Hydrated lime Filler No. 8	28
No. 24....	15 per cent Hydrated lime Filler No. 8	28
GROUP NO. II		
No. 25....	35 per cent Trap rock Filler No. 9	43
No. 20....	25 per cent Greenstone Filler No. 7	46
No. 32....	15 per cent Talc Filler No. 12	48
No. 43....	25 per cent Slate Filler No. 16	51
No. 4....	35 per cent Dolomite Filler No. 2	37
No. 7....	35 per cent Dolomite Filler No. 3	40
No. 10....	35 per cent Limestone Filler No. 4	43
No. 2....	25 per cent Slate Filler No. 1	45
No. 15....	15 per cent Slate Filler No. 5	43
No. 28....	15 per cent Supercel Filler No. 10	40
No. 33....	35 per cent Silica Filler No. 13	35
No. 36....	35 per cent Silica Filler No. 14	35
No. 39....	35 per cent Silica Filler No. 15	35
No. 40....	25 per cent Silica Filler No. 15	35
No. 47....	15 per cent Slate Filler No. 17....	43
No. 51....	35 per cent Limestone Filler No. 19	58
No. 52....	25 per cent Limestone Filler No. 19	48
No. 5....	25 per cent Dolomite Filler No. 2	28
No. 8....	25 per cent Dolomite Filler No. 3	37
No. 11....	25 per cent Limestone Filler No. 4	37
No. 26....	25 per cent Trap rock Filler No. 9	37
No. 3....	15 per cent Slate Filler No. 1	37
No. 21....	15 per cent Greenstone Filler No. 7	40
No. 34....	25 per cent Silica Filler No. 13	33
No. 37....	25 per cent Silica Filler No. 14	30
No. 44....	15 per cent Slate Filler No. 16	43
No. 48....	35 per cent Limestone Filler No. 18	45
No. 16....	35 per cent Sand Filler No. 6	28
No. 17....	25 per cent Sand Filler No. 6	28

The life or durability of the coatings in the accelerated weathering test are given in cycles (Table I). The results are the average of duplicate panels differing by not more than 3 to 5 cycles. It will be observed that in general the coatings failed in the same order in the outdoor tests as they did in the accelerated cycle. The end-point of the accelerated weathering test was determined by measurement of electrical conductivity. In the "conductivity test"[9] the panel is placed in a circuit and covered with a template having ten holes. The asphalt surface is then moistened through these holes with an electrolyte. If cracks through to the aluminum are present in the coating, the electrolyte will conduct electricity. The end-point of the test is reached when at least six of the ten holes in the template conduct current.

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[9] Method described in Proposed Method for Accelerated Weathering Tests on Bituminous Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, pp. 384–385 (1933).

Experience showed that while the conductivity test is satisfactory for testing panels exposed to the accelerated cycle, for which the test was designed, it fails in reliability when testing panels which have been exposed outdoors. In outdoor exposures the cracks in the coating fill with dust, which prevents the penetration of the electrolyte to the aluminum panel.

In the outdoor exposures the silica panels received higher ratings, when classified according to appearance, than in the accelerated cycle. The conductivity test showed that the panels exposed to the accelerated cycle were porous and absorbed the electrolyte readily, giving an end-point of test, although their appearance was still excellent. The relatively poor adhesion of the silica particles to asphalt is probably the cause of this porosity. In the case of the mixtures containing silica, therefore, classification in Table III by appearance is in some cases erroneous.

In the accelerated test, initial cracking appeared in all the unfilled coatings sooner than in any of the mixtures, but many of the mixtures reached the end-point of the test in the same number of cycles as the unfilled asphalts. This was particularly true of coatings containing 15 per cent of filler. In no case, however, did a filled coating prove less durable to weathering than an unfilled one. As a rule, within the range of filler content used in these tests (15 to 35 per cent) the greater the amount of filler the greater the durability. From this it follows, too, that the higher the fineness factor of a filler the less amount of material is required to obtain a given effect and for this reason the 15 per cent of mica is as efficient as 35 per cent of most of the other fillers.

Although these weathering tests show that all the fillers tested did not prove equally effective in enhancing the durability of the coating asphalt, no general conclusions can be drawn from the data as to the superiority of one type of filler over another. To obtain such information would necessitate a more systematic study, taking into account the interrelated effects of the composition of the filler, the state of subdivision and the ratio of the filler to asphalt, either by weight or by volume. By comparing the panels exposed outdoors with those exposed to the accelerated cycle it can reasonably be concluded that 30 cycles in the accelerated test are equivalent to about 1½ yr. outdoors.

Additional accelerated tests were made, first, using the same type of panels at less intense illumination, and second, using specimens coated on a saturated felt base instead of aluminum, with the less severe exposure to light. In both series of tests the order of failure was the same as that reported. Hence, it can be assumed that the tests described in this paper, using the aluminum panels, give results similar to those which would be obtained with regular roofing materials.

DISCUSSION

Mr. K. G. Mackenzie[1] (*presented in written form*).—Considering the widespread use of filler in coating asphalts used in roofing manufacture, the information presented in Mr. Strieter's paper is particularly timely. The amount of work required in an investigation of this problem is so extensive that it has been difficult to cover all possible combinations of mineral fillers and asphalt, but we believe that the various fillers commercially available have been tested quite thoroughly when used with an asphalt from one source.

It would be very desirable to continue this research using asphalts from other sources. This information might show that some fillers are peculiarly adaptable to certain asphalts and of little value when used with others. The need for scientific methods of evaluation of asphalts intended for roofing products is very evident.

I should like to express one word of caution in interpreting the results reported. Upon casual examination of Table III of the paper, one might conclude that roofing being manufactured today with the fillers reported in group No. I would weather two to two-and-one-half times as long as those manufactured with fillers from group No. III. Although the results of the accelerated weathering tests are in this ratio, we question if this ratio holds good in finished prepared roofings being marketed today.

Mr. Milton R. Beasley.[2]—Mr. Strieter's data would indicate that asphalts filled with practically any type of inert filler are superior to the non-filled asphalt and that is largely true. It should be kept in mind, however, that where asphalts are used for roofing purposes, there are other factors which should be considered, such as the effect of filler on the retention of roofing granules or the ability of the asphalt permanently to bond to the roofing granules.

Another point that should be considered is the shock resisting properties. Some fillers have greater ability to impart shock resisting qualities to an asphalt than others. I believe Mr. Strieter has previously found this out, but he did not mention it in this paper.

Mr. T. H. Rogers.[3]—Is the listing of the panels within groups Nos. I, II, or III in the order of best to worst, or is it simply haphazard?

Mr. O. G. Strieter.[4]—In general, the panels are listed according to their durability within the group; that is, the first panels in a group run better than the last panels.

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A FURTHER STUDY OF THE HETEROGENEITY OF ASPHALT—A QUANTITATIVE METHOD

By G. L. Oliensis[1]

Synopsis

This paper comprises a further discussion of the significance of the spot test, which test was first described before the Society by the author in 1933.[2] Several additional causes of heterogeneity are explained, other than high temperatures. The effectiveness of certain homogeneous asphalts in "correcting" heterogeneity is discussed. A method of determining heterogeneity quantitatively is presented, the procedure being the same as in the qualitative determination, except that instead of straight naphtha, mixtures of naphtha and xylene are used, the minimum proportion of xylene necessary to yield a negative "spot" determining the degree of heterogeneity quantitatively.

Steps Taken Towards Standardization of Test Since 1933

Since the presentation of the paper before this Society by the author in 1933[2] the test has been incorporated in a number of state, municipal, and industrial specifications. While some of the details of the test are occasionally modified, the procedure most widely recognized is the one officially adopted at the Third Regional Meeting of State Highway Engineers in Chicago, November 1, 1934, as appended hereto.[3]

It will be noted that that method is essentially the same as that first proposed in the paper referred to, differing only in that the specification for the naphtha is made more rigid and includes an aniline-number requirement, a No. 50 Whatman filter paper is specified for the spot, and a few other modifications are introduced to adapt it to the testing of "cut-back" asphalts.

No further change has been made in the procedure, though recent work by the author points to the desirability of substituting for the 25-ml. Erlenmeyer flask, a 25-ml. flat-bottom wide-mouth Soxhlet extraction flask, preferably of pyrex glass, because of the latter's slightly larger net volume, and its greater convenience in handling and in manipulating in the glycerine bath. It is also advisable to stipulate that when suspecting the presence of waxy bodies (which are indicated, for example, when a drop of the xylene solution on glass dries with a waxy sheen or nucleus), the solution, immediately before testing, should be warmed to 30 to 32 C., to prevent the waxy bodies from crystallizing out below that temperature and thus being mistaken for carbenoids.

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[2] G. L. Oliensis, "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

[3] See p. 506.

ORIGINAL CONCEPT OF SCOPE OF SPOT TEST

The test has been carefully studied in the laboratory of the U. S. Bureau of Public Roads, and in a number of other state, municipal, and industrial laboratories. To clarify some misconceptions that have arisen, and to define more concretely the logical scope and limits of the test as understood at the time the test was first presented, the following points are brought out:

1. The terms "homogeneity" and "heterogeneity" are used in a purely relative sense, and the dividing line established by the test between "homogeneous" and "heterogeneous" asphalts is therefore an arbitrary one, depending for its correct location on the use of a carefully standardized naphtha and a carefully standardized procedure.

2. The test has not been submitted as a quality test, but as an identifying test.

3. It will identify all petroleum asphalts that have been subjected to temperatures higher than are normal in the regular steam-refining processes. Hence it will detect (*a*) cracked asphalts, (*b*) steam-refined or vacuum-process residuals that have been accidentally subjected to higher temperatures than are normal, and (*c*) the more highly blown asphalts that have been subjected to the higher temperatures for prolonged periods of time.

4. It will not differentiate:

(*a*) Native asphalts from petroleum asphalts.
(*b*) Oxidized or "blown" asphalts from "unblown" asphalts.
(*c*) Asphalts derived from different geographical sources.
(*d*) Asphalts derived from different refining processes, except as indicated in paragraph 3 above.

5. It may or may not detect blends of "heterogeneous" with "homogeneous" asphalts, depending upon the content of the former and its degree of heterogeneity.

6. It should not be applied to cut-backs or crude oils containing appreciable quantities of distillates, till all these distillates have been carefully removed by an appropriate distillation process.

7. It should not be applied to any asphalt naturally containing finely-divided non-bituminous matter insoluble in xylene, for example certain native asphalts, since such fine non-bituminous bodies mask the presence of carbenoids if any exist. If, therefore, an asphalt contains such xylene-insoluble matter (as is indicated by a "positive" spot formed by its solution in xylene), its reaction should not be reported "positive" unless it is known that the asphalt is a purely bituminous petroleum residual, in which case its spot does warrant classification as "positive" because the presence of

xylene-insoluble matter, being abnormal in such residuals, raises a presumption of heterogeneity within the meaning of the spot test.

OTHER CAUSES OF HETEROGENEITY

It will be noted that in the foregoing summary the only type of "heterogeneity" referred to is that due to overheating, cracking, or extreme blowing, in all of which very high temperatures are involved. Since the presentation of the test in 1933, however, further studies have revealed that heterogeneity within the meaning of the spot test may be introduced by one or more of the following *additional* factors (in which high temperatures may not be involved):

(*a*) Waxy bodies,

(*b*) Acid sludge bodies,

(*c*) The combination of incompatible fractions in so-called "synthetic" asphalts, and

(*d*) Exposure to the air.

Heterogeneity Due to Waxy Bodies:

Waxy bodies, determined as "paraffin scale," can be extracted from practically all asphalts, the quantity varying from a mere trace in certain asphalts and highly asphaltic crude oils, to as much as 6 or 10 per cent in the residuals of "paraffin-base" crude oils. It is recognized that this paraffin or wax content is often seriously altered in the preliminary laboratory distillation required by the test, and that no indication is given by the latter of the more liquid paraffinaceous bodies that may be present. It is known, too, that these waxy bodies are better tolerated by some asphalts than by others. For example, straight paraffin wax may be safely combined in nearly all proportions with certain asphalts (gilsonite selects); may combine safely in certain proportions with other asphalts (Mexican or Venezuelan residuals); and may not combine at all with still other asphalts (grahamite). If combined in other than the tolerated proportions, an apparently homogeneous blend may sometimes at first be effected, which on prolonged heating in the kettle will precipitate a heavy sludge or sediment. Even crude petroleum underground will frequently precipitate its waxy bodies under predisposing causes, unless it contains lighter fractions of relatively high solvent power; but even in the latter case, when these lighter fractions are eventually removed in the distillation process, the residual may display its low tolerance for its waxy bodies, in the oily, waxy scum that develops on its surface on standing.

When asphalts contain waxy bodies for which they have a sufficiently low tolerance, they may be expected to react heterogeneous to the spot test. Furthermore, when such asphalts are heated, the degree of heterogeneity may increase with remarkable rapidity until actual precipitation

TABLE I.—QUANTITATIVE FIGURES ON VARIOUS HETEROGENEOUS ASPHALTS.

Sample and Type of Asphalt					Mexican Equivalent, per cent	Gilsonite Equivalent, per cent	Xylene Equivalent, per cent
I. Cracked Residuals:							
No. 1.—Pressure tar from crude A					85 to 87	42 to 45	78 to 80
No. 2.—Flux from Mid-Continent crude B					90+	90+	.....[a]
No. 3.—85-Penetration Residuals from crude C					.....	86 to 90	72 to 75
No. 4.—Heavy Cut-Back from crude C					.....	75	74 to 83
No. 5.—20-Penetration. Blend of Cracked Residual and Blown, from Mid-Continent crude D					75	70+	61 to 70[a]
II. Overheated Steam-Refined Residuals:							
No. 6.—Residual from crude E, 150 penetration					.....	26 to 30	11 to 15
No. 7.—Residual from crude E, 10 penetration					76 to 85	61 to 65	50 to 54
No. 8.—Residual from crude F, 35 penetration					.....	35 to 40	6 to 10
No. 9.—Residual from crude G, 10 penetration					51 to 55	31 to 35	21 to 25
No. 10.—Residual from crude G, 10 penetration					.....	56 to 60	35 to 40
No. 11.—Residual flux from crude H					.....	26 to 30	.....[a]
III. Wax-Bearing Residuals:							
No. 12.—Vacuum flux, crude J					.....	5	6 to 10
No. 13.—Vacuum flux, crude K					56 to 65	16 to 18	25 to 30
Laboratory Mixtures of Paraffin Waxes and Steam-Refined Residuals, per cent							
		40-Penetration Residuals From					
	Wax[c]	Arkansas Crude	South American Crude	Mexican Crude[c]			
No. 14-a	75	25			.....	.....	16 to 20
No. 14-b	75		25		.....	.....	11 to 15
No. 14-c1	75			25	.....	.....	30 to 31
No. 14-c2	75			25	.....	.....	32 to 34
No. 14-c3	75			25 (Freshly prepared)	61 to 70	26 to 30	30 to 35
				(After 50 hr. at 210 F.)	.....	.....	64 to 69
				(After 83 hr. at 210 F.)	.....	.....	Xylene insoluble matter formed[a]
No. 14-c4	75			25 (Freshly prepared)	.....	.....	30 to 35
				(After 20 min. at 500 F.)	.....	.....	64 to 69
				(After 30 min. at 500 F.)	.....	.....	Xylene insoluble matter formed[a]
V. Sludge Asphalt:							
No. 15-a.—Homogeneous Smackover residual combined in laboratory with 3 per cent concentrated H_2SO_4					.....	40	70 to 88
No. 15-b—Homogeneous Mexican residual combined in laboratory with 3 per cent concentrated H_2SO_4					.....	.....	11 to 20
No. 16.—Acid sludge submitted by oil refinery					.....	.....	Xylene insoluble
V. "Reconstructed" Asphalt:							
No. 17.—Sample of 180 penetration submitted by oil refinery					85	41 to 46	29 to 30
VI. Weathered Liquid Asphalt (Exposed 15 Weeks):[b]							
No. 18.—Mexican base					.....	10−	10 to 20
No. 19.—Mid-Continent base					.....	5	.....
No. 20.—California base					.....	11 to 20	20 to 25

[a] The xylene equivalents on samples Nos. 2, 5, 11, and the finals on 14-c3 and 14-c4 were difficult to establish accurately because they contained fine matter insoluble in xylene.

[b] Weathered Samples furnished by Courtesy of Bureau of Public Roads.

[c] Straight paraffin wax and straight Mexican residual, each heated individually for 4 hr. at 500 F. (260 C.), react negative to spot test.

of relatively coarse xylene-insoluble carbonaceous bodies results. A striking illustration of this is shown in Table I, samples Nos. 14-c3 and 14-c4. As a blank, the paraffin wax and the Mexican residual referred to were each heated separately to 480 to 500 F. (250 to 260 C.) for various intervals up to a total of 4 hr. Though both hardened considerably and the paraffin darkened in color in the process, they reacted negative to the spot test after every interval to the end. A fresh, unheated portion of this same wax and Mexican residual was then melted carefully, and the two were combined in the proportion of 75 per cent by weight of the former to 25 per cent of the latter. This freshly prepared blend of wax and Mexican residual reacted positive to the spot test. After this compound had been maintained at 480 to 500 F. (250 to 260 C.) for barely 20 min., its heterogeneity increased considerably, though it still remained completely soluble in xylene. After an additional 10 min. at that temperature, xylene-insoluble bodies began to form copiously at the sides and bottom of the dish, rendering further measurements of the increase in heterogeneity impractical (see Table I, sample No. 14-c4).

A fresh blend of the two was made up in the same proportions as before and maintained for 50 hr. at 210 F. (100 C.). Again a marked increase in heterogeneity was noted, followed, after an additional 33 hr. at the same temperature, by the formation of xylene-insoluble bodies (see Table I, sample No. 14-c3).

Much lower proportions of wax than above given are sufficient to develop heterogeneity, but the proportions were purposely set high so as to intensify the reactions and thus permit a clearer analysis of them.

The fact that the wax and the Mexican residual, homogeneous at the start, can be heated individually to 480 to 500 F. (250 to 260 C.) for long periods and remain homogeneous, whereas a blend of the two that is heterogeneous at the start becomes rapidly more heterogeneous on heating at the same or at much lower temperatures, appears exceedingly significant. A somewhat similar situation is revealed in two recent reports by the Bureau of Public Roads,[4] from the first of which the following statement is quoted:

> "Evidently carbonization, which takes place when bituminous materials are subjected to high temperature and which progresses more rapidly when they are subjected to sunlight, is intensified when the materials initially have free carbon and carbenes together in appreciable amounts."

It appears, in short, that once an asphalt has become heterogeneous as a result of the presence of waxy bodies, its heterogeneity may under certain conditions become aggravated by refining processes that are entirely safe for homogeneous asphalts, and a condition somewhat similar, in fact, to "cracking" may be induced in it by temperatures much lower than would cause "cracking" ordinarily.

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[4] "A Study of Some Liquid Asphaltic Materials of the Slow-Curing Type," *Public Roads*, Vol. 15, No. 4, June, 1934, p. 85; and "Further Studies of Liquid Asphaltic Road Materials," *Public Roads*, Vol. 16, No. 6, August, 1935, p. 97.

Heterogeneity Due to Sludge Bodies:

When concentrated sulfuric acid attacks a homogeneous bitumen, the acid sludge formed will cause the bitumen to react positive to the spot test, as long as traces of acid sludge are present.

Heterogeneity in "Reconstructed" Asphalt:

The hard, asphaltene-like bodies precipitated from crude oil by very light solvents like propane may be blended with fluxes, or with fractions derived from a further selective solvent process, to make what may be termed a "reconstructed" asphalt. Such reconstructed asphalt will react negative to the spot test if the blending flux or fraction has a sufficiently high solvent power towards the base; but will react positive to the spot test if it has too low a solvent power and is therefore incompatible with the base.

Heterogeneity Due to Weathering:

In the August, 1935, issue of *Public Roads*[5] an extensive study of the spot test on various liquid asphalts is described. Among other things, attention is called to the significant fact that ten samples of liquid asphalts that had tested negative to the spot test when fresh, had been found to test positive after they had been exposed to the sun for 5 weeks in films $\frac{1}{8}$ in. thick, carefully protected from dust and rain.

The author secured from the Bureau of Public Roads three samples of each of three originally homogeneous liquid asphaltic road materials, each of differing origin, and each of which had been exposed outdoors in the summer of 1934 for 5, 10, and 15 weeks in films $\frac{1}{8}$ in. thick, exactly as described in the preceding paragraph. All these nine samples of exposed asphalt were found by the author to react positive to the spot test, the degree of heterogeneity differing in the samples from the different crude oils but in all cases increasing progressively with the duration of the exposure. At the same time, the xylene solutions made with the nine samples all reacted negative. This proved that the positive reaction noted in the naphtha solutions was not due to fine, foreign matter that might have been suspected of settling into the films in the course of their exposure, but to the development of definite heterogeneity within the meaning of the spot test. This clearly corroborates the findings of the Bureau of Public Roads.

However, two samples of homogeneous vacuum-process residual of 40 penetration that had been exposed outdoors in $\frac{1}{8}$-in. films in November, 1935, at Madison, Ill., on a roof close to the author's laboratory, had not developed heterogeneity by the end of March, 1936, after five months' exposure.

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[5] Bureau of Public Roads, "Further Studies of Liquid Asphaltic Road Materials," *Public Roads*, Vol. 16, No. 6, August, 1935, p. 97.

There is not necessarily conflict between the results shown by the Washington and Madison samples, because the destructive action of the sun in winter is much less pronounced than in summer. Nevertheless, it is quite possible that the difference in results springs from another cause: namely, that the Washington samples are cut-backs and the Madison samples are solid residuals, and the internal structure of the cut-back may conceivably be the nearer of the two to the critical point where marked polymerization during weathering may occur.

Summary of Discussion of Causes of Heterogeneity:

It is seen from the foregoing that four distinct types of heterogeneity may exist in asphalts, beside the ones described in the earlier paper on the subject. However, of them all, only the type induced by the presence of waxy bodies seems likely to be encountered in the ordinary run of petroleum asphalt offered on the market, and in this type it is noted that a reaction similar to cracking may take place even in careful refining. Hence, the assertion in the earlier paper on heterogeneity, to the effect that the spot test identifies overheated or cracked products, may with propriety be made to apply also to asphalts containing this wax-induced type of heterogeneity, even though the latter may have been refined at normal temperatures.

METHODS FOR "CORRECTING" HETEROGENEITY

In the following discussion, references to "correcting" heterogeneity will be understood to mean merely "eliminating the symptoms of" heterogeneity, since there is as yet no way of determining whether any true corrective action is involved at all.

It has long been recognized that heterogeneity in asphalts can be corrected by the addition of other more homogeneous asphalts. Even before the spot test was released in 1933, other laboratories working independently had found that the addition of certain uncracked asphalts to cracked residuums reduced the latter's insolubility in carbon tetrachloride to less than 1 per cent.[6] At that time an insolubility of less than 1 per cent in carbon tetrachloride was the only criterion of homogeneity known to the industry, hence it was then claimed that the resulting blend was homogeneous. As a matter of fact, such a blend, even though showing less than 1 per cent insoluble in carbon tetrachloride, would not necessarily pass the spot test, and hence might still fail to qualify as homogeneous within the meaning of the latter test.

However, work done by the author as early as 1928 had already revealed that blends of heterogeneous and homogeneous asphalts could be made that would pass even the spot test, as ordinarily conducted on filter paper, provided that a sufficiently large proportion of an asphalt having a sufficiently safe margin of homogeneity were employed. Depending on whether an asphalt was only mildly heterogeneous or decidedly so, as little as one-twentieth of its volume to as much as ten to twenty times its volume of a highly homogeneous corrective asphalt would be required to make it pass the spot test.

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[6] U. S. Patent 2,024,096, Application February 23, 1932. Patent issued December 10, 1935. Assigned to The Texas Company, New York City.

Other laboratories, notably that of the Bureau of Public Roads (in 1934), have covered the same ground.

Practically any positively homogeneous asphalt will serve to "correct" heterogeneity by this process of blending, provided enough of it is used; but there is naturally a wide difference in the effectiveness of various asphalts in that direction. By far the most efficient asphalt so far found for that purpose is gilsonite (selects grade)—an asphaltite of high melting point that is distinguished from all other asphaltites and other asphaltic residuals of equally high melting point by its negative reaction to the spot test, by its ready solubility in petroleum naphthas in practically all proportions, and by its exceptional ability to correct the troublesome type of heterogeneity that develops in wax-asphalt compounds, such as are used, for example, in the insulated-wire trade.

Since the quantity of gilsonite or other homogeneous asphalt required to eliminate the symptoms of heterogeneity increases with the degree of heterogeneity, a study was made to ascertain whether this did not offer a means of determining the degree of heterogeneity quantitatively.

Both gilsonite selects and a highly-homogeneous Mexican residual of 180 to 200 penetration were accordingly employed in a series of tests; each being blended in different proportions with various heterogeneous asphalts to determine the approximate proportion required to make the latter pass the spot test. The blending with the Mexican residual required no special precautions since it could be accomplished without difficulty at low temperatures. But as gilsonite selects have the relatively high melting point of 325 F. (165 C.) and may therefore be overheated or charred when melted by an inexperienced operator, a special blending process had to be developed for it that would eliminate overheating and could be readily duplicated by other laboratories. The procedure finally adopted was as follows:

Assuming a blend is desired of, say, 75 per cent by weight of the heterogeneous asphalt and 25 per cent of gilsonite, 1.5 g.[7] of the former is placed in a 25-ml. flask and cautiously melted over a hot plate till it flows out over the bottom and up about $\frac{1}{4}$ in. on the sides. It is then allowed to cool, and 0.5 g.[7] of the gilsonite (freshly powdered under a steel roll to a size all passing the No. 20 screen) is added and spread uniformly over the sample of asphalt. The flask is then immersed in a glycerine bath maintained at

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[7] If the specific gravity of the blend is other than 1.00, this weight should be changed so as to yield 2 ml. of the finished blend.

400 F. (205 C.) for 5 min., which is usually amply sufficient to blend the two uniformly, after which the fluid mixture is flowed up about $\frac{1}{2}$ in. on the sides to make certain that thorough blending has taken place.

The 2 ml. of the gilsonite mixture so made (or the 2 ml. of the Mexican mixture similarly flowed out over the bottom of the flask) is then set aside to cool, 10.2 ml. of the standard naphtha is added, solution is effected within 6 to 8 min. exactly as in the ordinary spot test, and the solution is then tested to ascertain whether it reacts positive within 24 hr. If it does, the entire test must be repeated with a greater proportion of the homogenizing asphalt until a negative spot is obtained.

The proportion of the homogeneous asphalt required to secure this is reported as the corresponding "equivalent" for that particular asphalt. Thus, if it is found that a certain heterogeneous asphalt must be blended with its own weight of Mexican residual to produce a homogeneous blend, and with only one-fourth of its weight of gilsonite selects to produce a similarly homogeneous blend, the heterogeneity of that asphalt is reported as having a Mexican equivalent of 50 per cent and a gilsonite equivalent of 20 per cent, and an appropriate description of the Mexican and the gilsonite is added. The exact equivalent, can, of course be determined as sharply as may be desired, but for ordinary purposes it is sufficient to express the equivalent as within a range of three or four points. Thus if a gilsonite equivalent of 36 to 40 per cent is reported, it is understood that the presence of 35 per cent of gilsonite in the blend is insufficient to assure a negative spot, but that 40 per cent is sufficient, and that the exact equivalent, therefore, lies somewhere within the range of 36 to 40 per cent of gilsonite.

In Table I will be found the Mexican and gilsonite equivalents as actually established for a number of widely different types of heterogenous asphalts, the list including samples having all the different types of heterogeneity that have been discussed here. Most of the samples represent actual commercial production, but some few of the more unusual types have been, as indicated, prepared in the laboratory. The commercial grades are derived from twelve different crude oils, including Mid-Continent (both east and west of the Mississippi), West Texas, Mexican, South American, and California crude oils, and were obtained from nine different refineries, so that the list is fairly representative.

It will be noted that, generally speaking, the Mexican equivalent is higher than the gilsonite equivalent. This is especially true in the case of asphalts whose heterogeneity is due to the presence of waxes. For example, sample No. 13, the heterogeneity of which is due to waxy bodies, has a Mexican equivalent of 56 to 65 per cent, and a gilsonite equivalent of only 16 to 18 per cent, or less than one-third of the Mexican. On the other hand, in the case of cracked or overheated asphalts, the gilsonite usually appears but little more effective than the Mexican, as illustrated in samples Nos. 2,5 and 7.

QUANTITATIVE TEST FOR HETEROGENEITY

In a general way the data given indicate that these equivalents roughly establish a quantitative figure for heterogeneity, since the percentage of gilsonite, for example, varies from as little as 5 per cent in sample No. 12 (the heterogeneity of which was very slight, as indicated by the faintness of the nucleus), to over 90 per cent in the cracked products (the heterogeneity of which is very marked). But since gilsonite apparently has a greater aptitude for correcting heterogeneity due to waxy bodies than have other asphalts (and any other homogeneous asphalt may conceivably have similar special aptitudes), the use of any particular asphalt may be likely to favor heterogeneity of one type over that of another, and when employed for determining the degree of heterogeneity quantitatively may at times yield misleading results.

And yet there appears to be a definite need for some reliable quantitative test for heterogeneity. The most direct method would appear to be the filtration of the 24-hr.-old naphtha solution and weighing the "gross" carbenoid residue, from which the weight of any non-bituminous matter present could be deducted after redissolving the precipitate in xylene or carbon disulfide and filtering out the pure carbenoids. While this method has been studied for several years, a number of practical difficulties still remain, the most serious one being the extreme fineness of the carbenoids characteristic of certain types of heterogeneity, which enables them to pass through the finest filter paper.

In the search, therefore, for a quantitative test that would avoid these complications, a quick and simple method was at last developed, somewhat analogous to that used in obtaining the gilsonite equivalent, but with the homogeneizing agent transferred from the asphalt to the solvent. In brief, the procedure for obtaining a quantitative figure for heterogeneity is to run the spot test exactly as in the standard procedure, but instead of employing 10.2 ml. of straight standard naphtha, exactly the same volume of an arbitrary mixture of xylene and standard naphtha is employed, each being first measured out accurately from a burette and well mixed before use. If the resulting solution, which should be completed within 6 to 8 min. as in the standard procedure, tests positive within 24 hr., a fresh solution is made, but with a larger proportion of xylene in the mixed solvent, the volume of xylene being increased in increments of 1, 0.5 or 0.1 ml. at a time, depending upon how sharp an end point is desired. The volume of the naphtha is in each case correspondingly decreased until a negative test is obtained at the end of 24 hr.

The minimum percentage of xylene by volume in the mixed solvent necessary to assure a negative spot for a given asphalt is reported as the xylene equivalent of that asphalt. As in the case of the gilsonite equivalent determination, the end point of the xylene equivalent can be established as

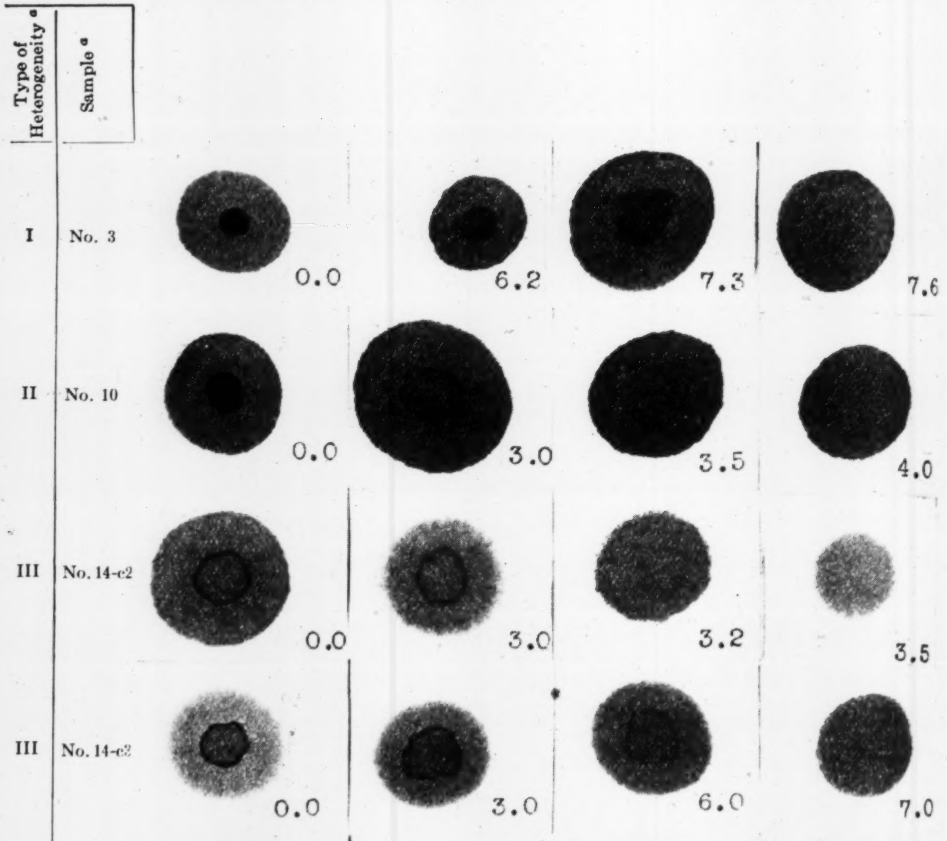
Type of Heterogeneity [a]
Sample [a]
I
No. 3
0.0
6.2
7.3
7.6
II
No. 10
0.0
3.0
3.5
4.0
III
No. 14-c2
0.0
3.0
3.2
3.5
III
No. 14-c3
0.0
3.0
6.0
7.0
IV
No. 15a
0.0
5.1
7.0
9.0
V
No. 17
0.0
2.7
2.85
3.0
VI
No. 20
0.0
1.0
2.0
2.5

FIG. 1.—Characteristic Spot Tests Showing Effect of Increase of Xylene in Naphtha-Xylene Solvent on Various Types of Heterogeneity.[b]

[a] For further identification of type of heterogeneity and type of asphalt, see corresponding number in Table I.
[b] The figure printed beneath each spot indicates the milliliters of xylene present in the 10.2-ml. solvent.

sharply as may be desired. For ordinary purposes a range of about three or four points seems sufficiently definite and can be so reported. Thus if 60 per cent xylene in the mixed solvent is not sufficient to overcome heterogeneity, but 65 per cent xylene is, the xylene equivalent may be reported as 61 to 65 per cent.

Needless to state, such asphalts as yield a positive reaction even in a straight xylene solution (and which are thus shown to contain fine non-bituminous matter and are therefore beyond the ordinary range of the spot test) cannot be tested for xylene equivalent accurately, though a rough estimate can no doubt be made in many cases by determining as closely as possible the minimum percentage of xylene required to reduce the intensity of the nucleus to the same degree as does straight xylene. However, since the petroleum asphalts on the market normally are pure bitumens and therefore normally test negative with straight xylene, all of them that do react positive to the spot test should readily lend themselves to an accurate determination of xylene equivalent.

In Fig. 1 are found a number of characteristic spots, illustrating the manner in which the nucleus is reduced in density and finally eliminated as the percentage of xylene is increased to the range of the xylene equivalent. No illustrations of the effect of the Mexican or gilsonite on the spot is included since it is quite similar to that of xylene.

In Table I is given the xylene equivalent for the twenty types of heterogeneous asphalt that have been so far examined, most of which have already been referred to in connection with the studies of Mexican and gilsonite equivalents. An analysis of these data and a comparison of the figures found in Table I, seem to warrant the following conclusions:

1. All the six different types of heterogeneity are characterized in common by their amenability to the corrective action of homogeneous asphalt or of xylene.

2. A comparison of the three types of equivalents indicates that Mexican is apparently too inefficient a homogenizer to be worth while as a quantitative standard; that gilsonite is apparently the most efficient of the three in correcting heterogeneity due to waxy bodies, and xylene the most efficient of the three in correcting heterogeneity due to cracking or overheating.

3. It is noted that the xylene equivalent reveals a much sharper differentiation than the gilsonite as between cracked residuals and overheated steam-refined asphalts; and since in actual physical and chemical characteristics these two types of asphalt are sharply differentiated from each other, the xylene equivalent probably presents a truer picture of the degree of heterogeneity than the gilsonite—which fact, together with the greater simplicity of its determination, warrants the consideration of the xylene equivalent as the most suitable of all three as a quantitative standard.

4. The range of accuracy of the xylene equivalent, as determined in the author's laboratory, is well within the four-point range suggested for reporting the test. Each equivalent is in fact self checking, since it consists of a series of trials in a steadily ascending or descending progression. Whether other laboratories, working with other supplies of solvent, will obtain equally satisfactory checks is yet to be determined.

5. A comparison of the different types of heterogeneity with one another indicates the type due to cracking to be the most aggravated of the six, since it corresponds to xylene equivalents of 60 to 80 per cent, and probably higher; while all the other types may apparently fluctuate from as low as 5 or 10 per cent xylene equivalent to as high as 50 per cent or more, but usually well below the latter figure. Apparently the heterogeneity induced in homogeneous asphalts by 15 weeks of outdoor summer exposure is of a relatively mild type.

6. Of special interest is the increase in xylene equivalent of the wax-Mexican blend, sample No. 14-c4, after heating 20 min. at 500 F. (260 C.), and of sample No. 14-c3 after heating 50 hr. at 210 F. (99 C.), and the formation of carbonaceous, xylene-insoluble bodies in both cases thereafter.

7. The fact that the spot test can now be used both qualitatively and quantitatively should greatly increase its effectiveness in differentiating asphalts of different degrees of heterogeneity, and in the intelligent control of refinery processes.

8. The fact that fluctuations in the degree of heterogeneity can now be measured quantitatively should give the research chemist a new tool for probing into the internal structure of asphalt, and determining how this structure may be affected in the various refining processes and under the destructive action of the elements.

APPENDIX

DESCRIPTION OF OLIENSIS SPOT TEST

FOR LIQUID ASPHALTIC MATERIALS OF THE SC, MC AND RC TYPES

Materials:

The naphtha solvent used shall be a straight-run overhead distillate free from cracked products of any kind, and shall conform to the following requirements:

Gravity, A.P.I.	49 to 50
Distillation	
Initial boiling point	Above 300 F. (150 C.)
50 per cent over	335 to 355 F. (170 to 180 C.)
End point	Below 410 F. (210 C.)
Aniline number[a]	138 to 145 F. (60 to 65 C.)

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[a] The aniline point of the solvent shall be determined as described in the A.S.T.M. Standard Method of Test for Precipitation Number of Lubricating Oils, (D 91 – 33), 1933 Book of A.S.T.M. Standards, Part II, p. 842.

Apparatus:

One 25-ml. Erlenmeyer flask.

Cork stopper for flask in which is fitted an 8-in. length of open $\frac{1}{4}$-in. glass tubing.

Filter paper—No. 50 Whatman.

Glass plate—smooth, clear glass, freshly cleansed with benzol or carbon tetrachloride, and soap-and-water then thoroughly rinsed with water, and wiped dry and free from dust or lint.

Sample:

For road oils of the SC class having less than 15 per cent by volume distilled off at 680 F. (360 C.) (by modified A.S.T.M. distillation test,[8] the test may be made on the original material. For all other materials of the SC, MC and RC classes, the test shall be made only upon the distillation residue.

Procedure:

A weight of the sample equivalent to 2 ml. is placed in the Erlenmeyer flask, and if it does not flow readily at room temperature it is cautiously heated in the flask on a hot plate till it flows out over the bottom. After cooling, 10.2 ml. of the solvent are added; the stopper, with its 8-in. length of tubing is quickly inserted into the neck of the flask; the contents of the flask are swirled with a rapid circular motion for 5 sec., and the flask is immediately immersed to its neck in a bath of gently boiling water (unless the sample is a thin liquid, in which case the heating bath is unnecessary). The contents of the flask shall again be swirled for 5 sec., at the end of each minute thereafter until complete dispersion takes place. If dispersion is not completed in from 6 to 8 min., the test shall be repeated with the bath maintained at a temperature 25 deg. Fahr. (14 deg. Cent.) lower or higher (glycerine being substituted for the water bath in the latter case) until dispersion is effected within the stipulated time. After complete dispersion, as judged by tilting the flask, the flask is allowed to cool to room temperature, and if any loss in weight occurs it is made up with additional solvent.

A drop of the asphalt solvent mixture is then placed on the filter paper. If the drop forms a brown or yellowish-brown circular stain, with a darker, solid or annular nucleus in the center, the test shall be reported as "*positive.*"

If, however, the drop forms a uniformly brown circular stain, judgment shall be reserved, and the asphalt solvent mixture shall be set aside in its tightly-stoppered flask at room temperature in a subdued light, for a further period of 24 hr. The mixture shall then be vigorously stirred till uniform, and a drop from it shall again be placed on filter paper. If the drop from the 24-hr. old mixture still forms a uniformly brown circular stain, the test shall be reported as "*negative*"; but if a darker, solid or annular nucleus, as described in the preceding paragraph now forms in the center of the stain, the test shall be reported as "*positive.*"

In case of dispute, the entire test shall be repeated, but only the residue from the modified A.S.T.M. test shall in that event be used. A drop of the asphalt-solvent mixture shall then be placed both on the filter paper and on the glass plate. If the appearance of the drop on filter paper (made either with the freshly prepared or the 24-hr.-old mixture) is still in dispute, then the test as made on glass shall be final. The test on glass is made in the following manner:

The drop shall be placed on the glass plate held at an angle of 45 deg. with the horizontal. If, as the drop flows out there develops in the center of its path a dull, matte streak, well defined against the smooth, clear glossy-brown outer zone, the test shall be reported as "*positive.*" But if a drop of the 24-hr. old mixture flows out to a uniform, clear, glossy-brown film, the test shall be reported as "*negative.*"

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[8] This modified method is substantially the same as the A.S.T.M. Tentative Method of Test for Separation of Liquid Asphaltic Products (D 402 - 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, 938 (1934); also 1935 Book of A.S.T.M. Tentative Standards, p. 855.

DISCUSSION

MESSRS. R. H. LEWIS[1] AND J. Y. WELBORN[1] (*presented in written form*).—Since the publication in 1933 of Mr. Oliensis' paper "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts,"[2] the U.S. Bureau of Public Roads has devoted considerable time to the study of this test which has proven to be a very useful one. The results of our studies corroborate the author's conclusions with respect to the scope and limitations of the test.

In our work on a large number of asphaltic materials from all producing centers it has been observed that heterogeneity is far more prevalent among the more fluid materials than among the refined semisolid paving asphalts, and the quantitative method developed by Mr. Oliensis should be of great value in comparing the degree of heterogeneity in various asphaltic materials. Since the release of the paper describing this new method of test we have had an opportunity to give it some study and the data secured are presented in this discussion.

Clifford Richardson in "The Modern Asphalt Pavement," states: "In residual pitches at times some of the bitumen is found which is insoluble in cold carbon tetrachloride, and this is evidently due to the severe treatment which the material has suffered in the course of its production at very high temperatures. A determination of the amount is only valuable as an indication of the care which has been used in the preparation of such pitches." For years asphalt specifications, therefore, have required a high solubility in carbon tetrachloride as a protection against overheating in the refining process.

However, in the examination of fluid and semisolid asphalts from numerous sources it has been found that many of the products which have been subjected to temperatures much higher than those used in normal steam-refining have a solubility in carbon tetrachloride which is relatively high. Many of these materials that are definitely cracking coil residues produce a positive stain with both naphtha and xylene, while the majority of those which apparently have been inadvertently overheated in a steam or vacuum process, and some blends of cracked and uncracked residuals, give a positive stain with naphtha and a negative stain with xylene. In spite of their high solubility in carbon tetrachloride, these asphaltic materials are heterogeneous. It is apparent, therefore, as has been pointed out by Oliensis, that a high solubility in carbon tetrachloride does not indicate definitely that the material has not been overheated in the refining process.

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[1] Associate Chemist, and Junior Highway Engineer, respectively, Division of Tests, U. S. Bureau of Public Roads, Washington, D. C.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

The Bureau is now engaged in a detailed investigation of commercial grades of semisolid asphalts from the refineries of the major producers. This study involves among other things the exposure of the asphalts in films $\frac{1}{8}$ in. thick to the action of sun and light for 15 weeks (approximately 900 sunlight hours).

TABLE I.—OLIENSIS SPOT TEST AND GILSONITE AND XYLENE EQUIVALENTS FOR 85 TO 100 PENETRATION ASPHALTS.

Sample	Base Petroleum	Refining Process	Tests Before Exposure			Tests After Exposure 15 weeks—$\frac{1}{8}$-in. Film	
			Standard Naphtha Stain	Gilsonite Equivalent, per cent	Xylene Equivalent, per cent	Standard Naphtha Stain	Xylene Equivalent, per cent
No. 1	California	Steam distillation	Negative	......	....	Positive	0 to 2
No. 2		Vacuum distillation	Negative	......	....	Positive	0 to 2
No. 3		Steam distillation	Negative	......	....	Positive	12 to 16
No. 4		Vacuum distillation	Positive	0 to 1	0 to 2	Positive	12 to 16
No. 5	Mexican	....	Positive	50 to 60	24 to 28	Positive	28 to 32
No. 6	Venezuelan	Steam distillation	Positive	5 to 8	2 to 4	Positive	12 to 16
No. 7		Continuous vacuum steam distillation	Negative	....	....	Positive	12 to 16
No. 8		Batch steam distillation	Positive	5 to 10	2 to 4	Positive	20 to 24
No. 9		Steam distillation	Positive	5 to 10	2 to 4	Positive	12 to 16
No. 10		Vacuum distillation	Negative	....	....	Positive	0 to 2
No. 11		Steam distillation	Negative	....	....	Positive	0 to 2
No. 12	Smackover	Vacuum distillation	Positive	0 to 1	0 to 2	Negative	....
No. 13	Oklahoma	....	Positive	30 to 35	44 to 48	Positive	72 to 76
No. 14		....	Positive	2 to 4	2 to 4	Positive	4 to 8
No. 15		....	Positive	5 to 10	4 to 8	Positive	8 to 12
No. 16		....	Positive	60 to 80	xylene insoluble	Positive	xylene insoluble
No. 17	Blend of Mexican and Oklahoma	Steam distillation	Negative	....	....	Positive	4 to 8
No. 18	Kansas	Winkler-Kock shell still	Positive	80 to 90	xylene insoluble	Positive	xylene insoluble
No. 19	....	Steam distillation	Positive	15 to 25	12 to 16	Positive	32 to 36
No. 20	....	Steam distillation	Positive	0 to 2	0 to 2	Negative	....
No. 21	Blend of Mexican and Domestic Gulf Coast Crudes	....	Negative	....	....	Positive	0 to 2
No. 22		....	Negative	....	....	Positive	2 to 4
No. 23	Texas	....	Positive	35 to 40	16 to 20	Positive	36 to 40

Thirty-nine samples of 85 to 100 penetration asphalt have been studied of which sixteen samples gave negative stains before and after exposure. Nine gave negative stains before exposure and showed varying degrees of heterogeneity after exposure. Twelve gave positive stains before and after exposure; and two materials that showed slight heterogeneity before exposure produced negative stains after 15 weeks of exposure. Perhaps these two are examples of the fugitive type which has been observed by Mr. Oliensis.

For the 23 samples which gave positive stains either before or after exposure, the accompanying Table I gives the base petroleum and the refining process as reported by the manufacturers, the results of the Oliensis spot test before and after exposure, the gilsonite and xylene equivalents before exposure and the xylene equivalent after exposure.

A study of Table I shows that only two of the 39 materials produced a positive stain with xylene, either before or after exposure. Eight of the 14 original materials which were heterogeneous had xylene equivalents of 8 or less, and gilsonite equivalents of 10 or less. Of the heterogeneous exposure residues of the 9 materials which were originally homogeneous, 7 had xylene equivalents of 8 or less. For those materials that were heterogeneous originally, the increase in xylene equivalent in the exposed residues is quite variable.

Samples Nos. 7 and 8 are of special interest. Both were produced from the same Venezuelan crude, the first by the continuous vacuum steam distillation and the latter by batch steam distillation. After exposure sample No. 8, which gave a positive stain initially, had a higher xylene equivalent than sample No. 7, which gave a negative stain initially.

In Table I of Mr. Oliensis' paper, he shows that the xylene equivalent for sample No. 13, representing a wax-bearing residual, crude K, is greater than the gilsonite equivalent. Sample No. 23 of our materials is a wax-bearing residual, produced from a crude oil that tests positive in the spot test. Tests made on other semisolid products from this same refinery and on residues reduced in the laboratory with a high steam ratio from the crude oils used as base petroleum by this refinery, required approximately 35 per cent gilsonite to produce a negative stain. Although the other samples from this source were not tested for xylene equivalent, it is noted that the xylene equivalent for sample No. 23 is considerably lower than the gilsonite equivalent. This is not in agreement with the conclusion of Oliensis that gilsonite is more efficient than xylene in correcting heterogeneity due to waxy bodies.

The spot test results made on these representative asphalts show that 23 out of 39 samples, or about 60 per cent, were homogeneous, and that 16, or about 40 per cent, remained homogeneous after 15 weeks' exposure. All of the homogeneous materials that became heterogeneous upon exposure had a xylene equivalent of 16 or less. Under the climatic conditions existing during the winter months at Madison, Ill., it is possible that heterogeneity might not have developed in these samples.

Under the same summer conditions to which the 85 to 100 penetration asphalts were subjected all fluid materials that we have studied developed heterogeneous residues within 5 weeks. In the report covering our study of the spot test on liquid asphalts, mentioned in Mr. Oliensis' paper, the authors made the following statement:

"Since the results of the tests were based upon the appearance of the spot as interpreted by the observer, it is difficult if not impossible to distinguish between border-line materials or to express clearly the apparent degree of heterogeneity that may be indicated by the varying degrees of non-uniformity in the stain. The classification given should be understood to mean that in the judgment of the observers, the materials and their residues gave stains that appeared either entirely uniform throughout or were only slightly non-uniform, having a slightly darker, more pronounced center, or else they had a definite dark to black center surrounded by a uniformly lighter stain, and were classified homogeneous, slightly heterogeneous, and heterogeneous, respectively."

The use of the xylene equivalent gives promise of overcoming past difficulties in the proper interpretation of the spot test and appears to offer a ready means of determining the comparative degree of heterogeneity and the rate of its development in materials exposed to weathering.

In a report presented at the January, 1936, meeting of the Association of Asphalt Paving Technologists[3] the behavior of 5 materials, 3 steam distilled residuals, Mexican, Mid-continent and California, and 2 cracking coil residuals under exposure conditions was described. The results of the spot tests, determined on these materials exposed in films of $\frac{1}{8}$, $\frac{1}{16}$, and $\frac{1}{32}$ in. thickness for 5, 10 and 15 weeks, may help to show the possibilities of the quantitative method of determining heterogeneity suggested by Mr. Oliensis.

TABLE II.—XYLENE EQUIVALENTS OF RESIDUES OF SLOW-CURING LIQUID ASPHALTIC MATERIALS (SC-2 GRADE) AFTER EXPOSURE.

	Sample No. 1 Mexican Residual			Sample No. 2 Mid-Continent Residual			Sample No. 3 California Residual		
	1/8 in. Film	1/16 in. Film	1/32 in. Film	1/8 in. Film	1/16 in. Film	1/32 in. Film	1/8 in. Film	1/16 in. Film	1/32 in. Film
After 5 weeks exposure	4 to 8	8 to 12	12 to 16	0 to 2	28 to 32	48 to 52	24 to 28	36 to 40	48 to 52
After 10 weeks exposure	0 to 2	4 to 8	xylene insoluble	16 to 20	36 to 40	60 to 64	32 to 36	44 to 48	64 to 68
After 15 weeks exposure	8 to 16	xylene insoluble	xylene insoluble	36 to 40	56 to 60	68 to 72	36 to 40	48 to 52	xylene insoluble

The two cracking coil residues gave positive spots with xylene, and the development of heterogeneity in these materials under exposure could only be detected by the steady development of organic matter insoluble in CS_2 and CCl_4. The other three materials and their distillation residues gave negative stains. The xylene equivalents of the residues after various periods of exposure are shown in the accompanying Table II.

It will be seen that at each test period the thinner the film thickness the greater the xylene equivalent. For each thickness of film, except the $\frac{1}{8}$- and $\frac{1}{16}$-in. samples of the Mexican residual, the xylene equivalent increased with the time of exposure. The reason that the $\frac{1}{8}$- and $\frac{1}{16}$-in. films of the Mexican residual at 10 weeks have lower xylene equivalents than the corresponding 5 weeks' films is not known.

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[3] A Report on the Weather-Resistant Properties of Certain Slow-Curing Liquid Asphaltic Materials, *Proceedings*, Assn. Asphalt Paving Technologists, January 23, 1936, p. 1.

The following data with respect to the solubilities of some of these residues are of interest:

SAMPLE	TIME OF EXPOSURE, WEEKS	FILM THICKNESS, IN.	TOTAL ORGANIC INSOLUBLE IN CS_2, PER CENT	TOTAL ORGANIC INSOLUBLE IN CCl_4, PER CENT	XYLENE EQUIVALENT
No. 1.........	10	$\frac{1}{32}$	1.88	1.98	Insoluble
No. 1.........	15	$\frac{1}{16}$	1.42	1.67	Insoluble
No. 1.........	15	$\frac{1}{32}$	2.88	3.22	Insoluble
No. 2.........	15	$\frac{1}{32}$	1.65	2.06	68–72
No. 3.........	15	$\frac{1}{32}$	1.21	1.40	Insoluble

It will be seen that the $\frac{1}{32}$-in. film of sample No. 2 after 15 weeks' exposure has more matter insoluble in CS_2 than 2 of the other residues, and more matter insoluble in CCl_4 than 3 of the other residues. However, it is readily dispersed in 68 to 72 per cent xylene-naphtha solution, while the other samples all contain material insoluble in 100 per cent xylene. This would seem to indicate a difference in the character of the insoluble materials in the several residues which affects their dispersion in the solvents used for determining heterogeneity.

The stain obtained in the original Oliensis spot test indicates only that the material is homogeneous or heterogeneous. The degree of heterogeneity is not determinable and, as indicated in Table I, it automatically classes materials which have been only slightly overheated with products which have been subjected to excessive heat. This quantitative method proposed by Mr. Oliensis, therefore, should prove extremely valuable in future studies of asphaltic materials.

MR. J. M. WEISS.[4]—It has occurred to me that the mixture of wax and asphalt heated at temperatures way below anything we know causes cracking may involve a reaction similar to that we experience with coal tars and benzol where, if we let them stand, the content of extremely high boiling hydrocarbons which are insoluble, increases regularly with the time they are allowed to stand, even at room temperatures. It may be a combination of hydrocarbons in paraffin and the higher ones in asphalt resulting in a complicated hydrocarbon of higher molecular weight which becomes insoluble in the mixture and which might be studied, possibly by isolation and ultimate analysis.

MR. T. H. ROGERS.[5]—I note that it is stated that the development of quantitative features to the method makes it of particular value from a research standpoint. I think that is perhaps true. I do think, however, that from the standpoint of empirical and immediate use the test becomes perhaps less satisfactory than it was before, in view of the evidence of all these fine gradations and distinctions as to degree of heterogeneity. We have all kinds and degrees of material which for one reason or another may be more or less heterogeneous. The question arises—what does that all mean? The uncertainty is illustrated by the fact that the paper gives an example of what may be interpreted as cracking, which behavior resulted from heating a mixture of wax and residual for 50 hr. on a steam bath. Certainly from any other standpoint, considering the basic information on the cracking reaction, it is entirely erroneous to conclude that cracking takes place under those conditions.

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[4] President, Weiss & Downs, Inc., New York City.

[5] Assistant Director of Research, Standard Oil Co. (Indiana), Whiting, Ind.

As a specific question, with reference to interpretation, I should like to inquire what would be the conclusions if the conditions of the test are appreciably modified. All of the present work depends on tests with a certain kind of naphtha in a certain ratio to the oil. Presumably all of these fine distinctions as to what is—or is not—heterogeneous would have to be modified if a different naphtha, or a different amount of naphtha, were employed.

MR. G. L. OLIENSIS.[e]—The remarks just made resolve themselves into three questions: Let me discuss the last one first, as it refers to the technique of the test as originally released three years ago: it raises the point whether, if the type of naphtha or its proportions were substantially altered, the spot test might not give altogether different classifications to many asphalts. This is to some extent true, and it is precisely for that reason that the details of procedure were made so rigid that the chances for divergent classifications in different laboratories might be reduced to a minimum. But it should be pointed out that numerous other tests have similar limitations. For instance, the naphtha-soluble test, so commonly used, would yield altogether different percentages of asphaltenes if the type and proportion of naphtha were altered.

However, there is still another angle to this question: namely, whether the standard type of naphtha and the standard proportions used in the spot test were selected in a purely arbitrary manner, or have been established at a point where they can serve the best purpose. This is answered by the fact that under the standard procedure cracked or overheated asphalts fail to pass the spot test by a wide margin, while normal steam-refined or vacuum-processed asphalts similarly pass by a wide margin. Between these two groups there is a region in which but relatively few asphalts are found, and right through that "no man's land" runs the line of demarcation that has been established in that test. As a result, while there have been undoubtedly thousands of samples subjected to the spot test in the past three years, there have been encountered in that time probably no more than half a dozen samples that were so close to the border line that different laboratories had difficulty in reaching an agreement as to their classification. In every such case, by a painstaking adherence to all details of the standard procedure, I was able to find that the asphalt in question either definitely passed or definitely failed to pass, and to check my conclusions repeatedly. Even these semi-doubtful cases, however, were so few, that I am encouraged to say that if the type and proportions of naphtha were modified slightly, the classification of the large bulk of the asphalts sold on the market would not be altered, but a good deal of confusion and controversy in the classification of those relatively few asphalts in the so-called "no man's land" would be introduced.

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[e] Chemist in Charge, Technical Bureau, The Barber Asphalt Co., Madison, Ill.

The second question that has been raised is whether I was warranted in making the statement in my paper that some type of cracking occurred in a mixture of asphalt and paraffine wax that was heated for 50 hr. on a steam bath. It will be recalled that after 83 hr. free carbon was formed in this mixture. I believe that regardless of what meaning is usually applied to the term "cracking" in oil refinery practice, it will be generally held in the asphalt industry that when a heat reaction takes place in a pure-bitumen asphalt that develops in it substantial quantities of coked material or free carbon, totally insoluble in carbon bisulfide, this constitutes essentially a cracking reaction.

The third question is raised by the remark made by Mr. Rogers that the quantitative method makes the spot test in practical work perhaps less satisfactory than it was before. It should be borne in mind that the quantitative method was submitted mainly for its usefulness to the research worker and plant chemist. The former may be interested, for example, in studying the gradual development of heterogeneity in different asphalts exposed to the same weathering cycle; the plant man may wish to determine the rate of decline of heterogeneity as the refining temperatures are decreased, or the steam or vacuum increased. The quantitative method will help them both in such studies. However, I feel that there would be no point in adopting the quantitative method in current specifications as yet. The main use of the spot test in present specifications is to differentiate clearly between just two classes of asphalts—those that do not pass that test and those that do. For this the qualitative method seems entirely adequate, and it would therefore be superfluous to burden the purchaser's laboratory with the further task of determining exactly how far those asphalts have failed that have not passed the test.

MR. ROGERS.—I am very glad to get your point of view on that and may I raise one further point? I believe that, in a discussion about a year ago, someone remarked that the asphalt technologist would find a way of correcting the spot test of his asphalt and I gather this is of assistance to the refinery chemist doing that. I am wondering, though, what evidence we have that he is actually improving the service quality of his asphalt by correcting the spot test? He is not removing the material which originally caused the heterogeneity.

MR. OLIENSIS.—I can give but little information on this subject; that is precisely why, in discussing methods for correcting heterogeneity in my paper, I pointed out that I was using the term "correcting" merely as synonymous with the term "eliminating the symptoms of" heterogeneity, since there is as yet no way of determining whether any true corrective action is involved at all. However, our study of methods for correcting heterogeneity does show that very substantial proportions of a homogeneous asphalt must generally be added to a heterogeneous one to make the latter pass the spot test. Table I shows that to correct the heterogeneity of a steam-refined asphalt that was unintentionally overheated, from 1 to 5 times its weight of a homogeneous asphalt had to be added; while to correct the heterogeneity of a true cracked asphalt, as much as 5 to 20 times its weight of a homogeneous asphalt had to be blended with it. Naturally, as a greater and greater proportion of a homogeneous asphalt is introduced into a heterogeneous one, the greater will be the physical dilution of the heterogeneous bodies in the latter, and the greater the extent to which the resulting blend will tend to acquire the characteristics of the homogeneous asphalt; and to that extent, if no other, will its service characteristics probably be affected.

MR. LEROY M. LAW[7] (*by letter*).—Shortly after the publication of Mr. Oliensis' original paper, the proposed test for heterogeneity, with some slight modifications, was adopted by a group of highway testing engineers meeting in Chicago and, within a comparatively short time, it became incorporated in many state and municipal specifications. This rather unusual procedure was taken to meet an insistent demand for a test that would identify what we commonly understood as residual products obtained in connection with gasoline cracking processes and although such materials doubtless constitute a great majority of heterogeneous asphalts in the paving industry, future interpretations of the test must take into account that there are other types of materials which may be so classified.

As to heterogeneity due to waxy bodies, Mr. Oliensis' work appears to harmonize with my own experiences with some of the early Mexican crude oils. At least one of these raw materials contained notable percentages of paraffin constituents and, when tested in the laboratory according to the Holde procedure, showed from 3 to 5 per cent of paraffin scale depending upon conditions of the distillation. Similarly in the refinery, and until appropriate conditions were found and rigidly adhered to, these paraffin constituents would tend to go into crystalline form as the crude approached asphalt consistency. When freshly made these asphalts appeared and tested normal but in time they lost luster, developed waxy surfaces and later lost ductility and adhesiveness. Looking back over these experiences in the light of Mr. Oliensis' work it would appear that these asphalts had a relatively small tolerance for the waxy constituents in the form in which they were produced under conditions of the distillation. Modern processes and equipment of course tend towards the elimination of such conditions and yet it is conceivable that a crude oil might exist that cannot be refined under any practical conditions and at the same time yield homogeneous asphalt products.

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[7] Asphalt Technologist, Shell Petroleum Corp., St. Louis, Mo.

As to the quantitative feature of this paper, it seems to me that this is of lesser importance, except of course for research purposes than the study of the various causes or types of heterogeneity and methods for their identification.

Certainly until we know more as to the behavior of such materials under service or under accelerating tests, we shall be at a loss in setting any quantitative limits. However, in order to simplify Mr. Oliensis' method based on two solvents, xylene and naphtha, might it not be well to employ for instance six definite reagents containing 0, 20, 40, 60, 80 and 100 per cent xylene? These could be given the numbers 0, 1, 2, 3, 4 and 5 and, after making the "spot test" with all of them, the material under investigation could be given a corresponding heterogeneity number such as 0, 1, 2, 3, 4 or 5 depending upon which reagent showed the last negative test. I would be interested in knowing how a suggestion of this kind would appeal to Mr. Oliensis.

MR. OLIENSIS (*author's closure by letter*).—The trouble experienced by Mr. Law in refining the high-paraffin crude oil brings out strikingly the complications that may be introduced in an asphalt by the presence of waxy bodies and the possibility that reactions once started in an asphalt may keep on progressing even under normal atmospheric temperatures.

Mr. Law's suggestion for a simpler method of reporting quantitative tests on heterogeneity deserves careful consideration. At the present time, of course, when only the research and plant chemists are likely to be interested in quantitative work on heterogeneity, a simplified method is really not necessary, as each chemist will undoubtedly wish to decide for himself within what range of accuracy his records must be kept to be most useful to him. If in the future, however, our increasing knowledge of the practical significance of the spot test led us to believe that a product having a certain type of heterogeneity, or a certain degree of it, was particularly useful for a given service or process, then the time would be ripe for incorporating a quantitative requirement with the spot test in purchasers' specifications, and at such a time Mr. Law's suggestion for dividing the field of heterogeneity into six groups, or perhaps ten or more groups, each identified by a single number, would certainly simplify specification writing and lessen the likelihood of controversy between different laboratories.

Incidentally, an opportunity recently presented itself of determining how closely the xylene equivalents as obtained by different laboratories on the same sample may be expected to agree. The sample investigated was the 85-penetration cracked residual identified as No. 3 in Table I of the paper, and portions of that sample were submitted to three other laboratories. The following xylene equivalents were found:

Author's laboratory	Original report	72 to 75 per cent
	Recent test	73 to 74 per cent
Laboratory No. 1		76 to 77 per cent
Laboratory No. 2		71 to 75 per cent
Laboratory No. 3		70 to 74 per cent

Some confusion arose in the course of this collaborative work as to the exact manner of calculating the final percentage figures from the actual volumes of xylene used. The method contemplated in the paper is to convert the actual volume of xylene into percentage to the first decimal place, and report the *next higher* whole percentage in the case of the lower xylene equivalent; while for the higher xylene equivalent the *nearest* whole percentage is reported, a value of 0.5 per cent in the latter case being considered nearer the higher than the lower whole number. Thus if 8.0 ml. xylene yields a positive spot, and 8.4 ml. a negative spot, these two figures divided by 10.2 would be equal respectively to 78.4 and 82.3 per cent, and would be reported as 79 to 82 per cent xylene equivalent.

The close agreement between the four laboratories, as shown in the foregoing tabulation, is exceedingly encouraging, and seems to establish the practicability of the xylene-naphtha method for the quantitative determination of heterogeneity.

MEASUREMENT OF HIGH VISCOSITY—A RAPID METHOD

By R. N. Traxler[1] and H. E. Schweyer[2]

Synopsis

The determination of the viscosities of highly viscous materials presents problems and requires methods and apparatus not commonly used in viscometry.

Diagrammatic illustrations, directions for operating and formulas used for calculating the viscosities in absolute units are given for the following types of instrument:

1. The capillary tube viscometer modified to measure viscosities from 10 poises up to 1,000,000 poises.
2. The alternating stress method by means of which bituminous materials ranging in viscosity from 1,000,000 to 100,000,000 poises have been successfully measured.
3. The rotating concentric cylinder viscometer, which may be used to measure a wide range of viscosities.
4. The falling coaxial cylinder type, which can also be used over a wide range.

When considering the development of a rapid, accurate method for control purposes, the last-mentioned type of instrument seemed to offer several possible advantages over the other methods. Consequently, twenty-five different sizes of falling coaxial cylinder viscometers were studied in order to select the most satisfactory instrument for rapidly measuring viscosities above 5000 poises. This instrument has been adapted so that it can be used as a laboratory control viscometer for a very wide range of viscosities.

The information obtained by viscometric methods concerning the behavior and characteristics of highly viscous materials is very useful. However, the determination of the viscosities of such materials requires special methods and presents problems not usually encountered in viscometry. Bituminous materials are especially interesting because, although many of them are essentially viscous liquids, the characteristics associated with structural viscosity are frequently encountered during a study of the harder asphalts and filled bitumens at atmospheric temperatures. Among the properties encountered are elasticity, thixotropy, dilatancy (with highly filled bitumens) and varying degrees of non-Newtonian flow. Because of the presence of these properties it is frequently impossible to establish absolute values for the viscosities of the harder asphalts and highly filled bitumens.

For a number of years the measurement of high viscosities has been studied in our laboratories. Various methods have been tried and it has been found that each has its advantages and disadvantages under certain circumstances and for particular types of material.

[1,2] Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N.

Capillary-Tube Method:

Probably the best known form of viscometer is the capillary tube because much study has been given to this type of instrument in general viscometry.[3] Bingham and Murray[4] developed a tube apparatus which they employed for measuring the consistency of paints. Their instrument has been modified and critically studied[5] using paints and heavy oils. The same principle has been successfully adapted[6] to the measurement of viscosities from less than 10 poises up to 1,000,000 poises. A capillary-tube type viscometer by means of which viscosities up to 20,000 poises can be accurately measured has been developed by Rhodes, Volkmann and Barker.[7]

Figure 1 shows a diagrammatic sketch of the apparatus used in our laboratory. The viscometer is enclosed in a thermostatically controlled cabinet with a double-walled glass front through which the flow of the material along the tube can be observed. Frequently, the tubes used are too large to be called capillaries; one with an internal diameter as large as 1 cm. has been employed in the measurement of substances with viscosities of approximately 1,000,000 poises. The asphalt in the reservoir *A* is first brought to the desired temperature and then, by air pressure applied through tube *B*, it is forced out a short distance into the straight tube *C* of uniform bore. This tube is mounted in front of a millimeter scale. The compressed air used in moving the bitumen is drawn from a reservoir which provides constant pressure and the applied pressure is measured by a manometer *D*. Measurements are made of the time required for the asphalt or other material to advance a certain distance in tube *C* under a particular

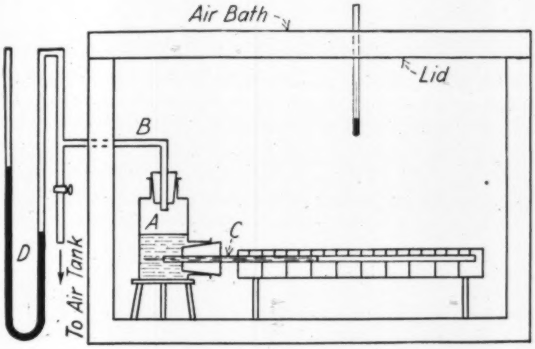

FIG. 1.—Apparatus Used in Capillary-Tube Method.

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[3] E. C. Bingham, "Fluidity and Plasticity," McGraw-Hill Book Co., Inc., New York City (1922).

[4] E. C. Bingham and H. A. Murray, Jr., "A New Combined Viscometer and Plastometer," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 655 (1923).

[5] D. V. Gregory, G. M. Rassweiler and K. C. Lampert, "A Modified Plastometer for Industrial Use," *Journal of Rheology*, Vol. 1, p. 30 (1929).

[6] C. U. Pittman and R. N. Traxler, "Rheological Properties of Asphalts. I. Effect of Temperature," *Physics*, Vol. 5, p. 221 (1934).

[7] E. O. Rhodes, E. W. Volkmann and C. T. Barker, "New Viscosimeter for Bitumens Has Extended Range," *Engineering News-Record*, Vol. 115, p. 714 (1935).

applied pressure. Successive readings are made with decreasing applied air pressures; thus viscosity values are obtained at different shearing stresses. The bituminous materials which are soft enough to be tested in this apparatus generally exhibit viscous flow and, consequently, anomalous flow characteristics are not encountered.

The viscosity is calculated by the equation

$$\eta = \frac{PR^2t}{8Ld} \qquad (1)$$

where η = viscosity in poises,
P = pressure applied in dynes per square centimeter,
R = radius of the tube in centimeters,
t = time of flow in seconds,
L = length of asphalt column in centimeters, and
d = distance of flow in time t.

In order to avoid end effects the asphalt is forced out into the tube for a short distance before any measurements are made. The column of asphalt L being sheared is varying continuously and is equal to the distance from the end of the tube at which the asphalt enters to the point mid way between the place where flow starts and stops for each measurement. At low shearing stresses account must be taken of the pressure due to the asphalt in reservoir A.

Alternating Stress Method:

An adaptation of the capillary-tube method was employed by Bingham and Stephens[8] to measure the viscosities of rosin, abietic acid and pitch at temperatures ranging from 20 to 60 C. The alternating stress method has been used successfully in our laboratories[6,9,10] for measuring bituminous materials ranging in viscosity from about 1,000,000 to 100,000,000 poises. The apparatus is illustrated diagrammatically in Fig. 2. A cylinder A of the material, about 5 cm. long, is cast in a glass tube B. This is accomplished by placing the tube B in a vertical position with an amalgamated brass plunger with a flat end inserted part way into the tube. The asphalt is poured into the tube and onto the plunger. After cooling, both ends of the plug of asphalt are flattened by manipulation with the amalgamated plunger. The internal diameter of the tube B may vary from about 0.25 to 2.00 cm. Tube B is connected to a tank of compressed air and a manometer C by three-way stopcocks D so that pressure can be applied to either end of the cylinder of bitumen. The tube B and its contents are placed in a constant temperature bath in such a way that the ends of the cylindrical sample can be observed through a cathetometer. A glass-bottomed pan is fastened in the top of the water bath to prevent the waves created by the stirrer interfering with the observations.

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[8] E. C. Bingham and R. A. Stephens, "The Alternating Stress Method for the Measurement of the Consistency of Very Stiff Materials," *Physics*, Vol. 5, p. 217 (1934).

[9] R. N. Traxler, C. U. Pittman and F. B. Burns, "Rheological Properties of Asphalt. II. Discussion of Penetration-Viscosity Relationships," *Physics*, Vol. 6, p. 58 (1935).

[10] R. N. Traxler and H. E. Schweyer, "Rheological Properties of Asphalts. III. A Viscosity Index," *Physics*, Vol. 7, p. 67 (1936).

The cathetometer is focused on one end of the cylinder and measured air pressure is applied to the other end. An extrusion of the material occurs, the rate of movement of which can be followed by the cathetometer. If the material is viscous and undergoes true laminar flow in the tube, the extrusion assumes the shape of a paraboloid of revolution. The extruded mass must not be permitted to wet the wall of the tube or be allowed to remain out so long that it slumps due to the action of gravity. In order to obviate slumping of the extrusion, tube *B* could be placed in a vertical position in a glass-walled water bath. This would necessitate a slight correction for the gravitational effect. Our technic has been to carry the extrusion out to a distance equal to one-half of the radius of the tube and force it back by reversing the direction of the application of the air pressure. High pressures are applied first and each succeeding pair of determinations is made at progressively lower pressures (shearing stresses). If the material is elastic or thixotropic the sample must be worked back and forth several times before a reading is taken. Materials which show marked dilatancy are not successfully measured by this instrument. Viscosity in poises may be calculated by the expression:

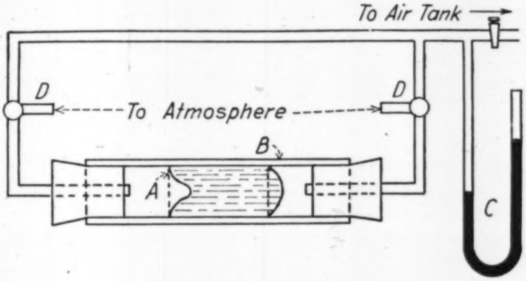

FIG. 2.—Apparatus Used in Alternating Stress Method.

$$\eta = \frac{PR^2t}{4Lh} \qquad (2)$$

where η, P, R and t have the same significance as in Eq. 1,
L = length of asphalt cylinder in centimeters, and
h = length of extrusion, in centimeters, in time t.

ROTATING CYLINDER METHOD

Recently, considerable interest has been shown in the rotating concentric cylinder type of viscometer for the study of highly viscous materials.

Saal and Koens[11] investigated the flow properties of asphaltic bitumen using the Couette[12] type of instrument. Mooney and Ewart[13] avoided the difficulties encountered due to end effects by making the ends of the rotating inner cylinder slightly conical in shape. They give a complete theoretical treatment of the instrument. Parks and his co-workers have used a rotating concentric cylinder viscometer to measure the viscosities of under-cooled liquid glucose,[14] commercial rosin and abietic acid,[15] boron trioxide,[16] and polyisobutylene.[17]

An instrument similar to that proposed by Mooney and Ewart has been employed in our laboratories to study the flow properties of bitumens possessing a wide range of consistencies. Figure 3 illustrates diagrammatically the essential parts of the apparatus. Outer cylinder A (the stator) is made so that it may be locked firmly in the thermostatically controlled water bath. The inner rotating cylinder B (the rotor) which is slightly conical at both ends is centered by means of hardened steel bearing points at both ends. We employed a stator 19 cm. high inside and with an internal

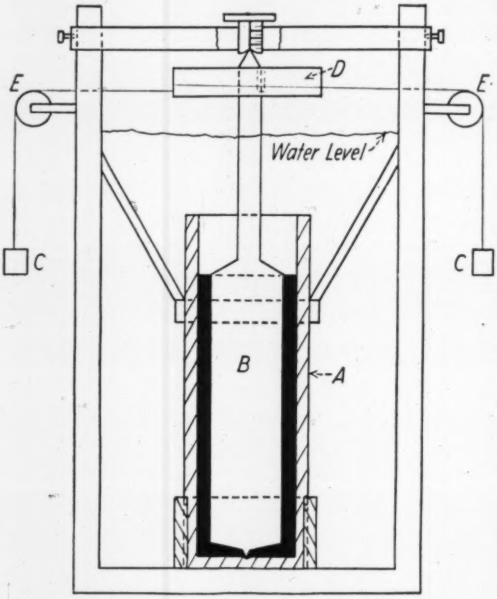

FIG. 3.—Apparatus Used in Rotating Cylinder Method.

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[11] R. N. J. Saal and G. Koens, "Investigations into the Plastic Properties of Asphaltic Bitumen," *Journal*, Inst. Petroleum Technologists, Vol. 19, p. 176 (1933).

[12] M. Couette, "Studies Concerning the Friction of Liquids," *Annales de chimie et de physique*, Vol. 21, No. 6, p. 433 (1890).

[13] M. Mooney and R. H. Ewart, "The Conicylindrical Viscometer," *Physics*, Vol. 5, p. 350 (1934).

[14] G. S. Parks, L. E. Barton, M. E. Spaght and J. W. Richardson, "The Viscosity of Undercooled Liquid Glucose," *Physics*, Vol. 5, p. 193 (1934).

[15] G. S. Parks, M. E. Spaght and L. E. Barton, "Viscosity Data for Commercial Rosin and Abietic Acid," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 7, p. 115 (1935).

[16] G. S. Parks and M. E. Spaght, "Some Viscosity Data for Boron Trioxide," *Physics*, Vol. 6, p. 69 (1935).

[17] J. O. Ferry and G. S. Parks, "Viscous Properties of Polyisobutylene," *Physics*, Vol. 6, p. 356 (1935).

radius of 2.540 cm. Our rotors varied in radius from 1.031 to 2.065 cm., thus giving annuli from 1.509 to 0.475 cm. in thickness. The cylindrical surfaces of the rotors were about 15.3 cm. long. Other sizes could be used and possibly would be more satisfactory for certain materials.

The inner cylinder is rotated by means of weights, *C*, attached to cords or piano wires wound in opposite directions on drum *D*, and passing over pulleys *E*. By allowing half the load to pull on each side of the rotor, the forces are distributed equally. Thus, the cylinders are kept concentric and the friction of the instrument is minimized. The rate of rotation is measured by means of a metal protractor clamped to the top of drum *D*.

To fill the viscometer the outer and inner cylinders are removed from the water bath, heated to about 110 C. in an oven, and molten asphalt poured into the space between them. Before the asphalt has an opportunity to cool appreciably the viscometer is put back into the frame which is an integral part of the water bath. Thus, the cylinders are made concentric with asphalt filling the annulus. After sufficient time (1 to 2 hr.) has elapsed for the bitumen to come to the temperature of the bath, equal loads are applied to both wires and the rates of rotation determined. A number of different loads are usually employed in order that the viscosity may be obtained at different shearing stresses. The viscosity is calculated by the expression:

$$\eta = K\frac{T}{\omega}\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

where η = viscosity in poises,
T = total torque applied to rotor,
ω = angular velocity of rotor in radians per second, and
K = a constant involving the dimensions of the instrument.

The equations for the evaluation of the constant *K* are given in detail by Mooney and Ewart.[13]

The friction in this viscometer is so small that it may be neglected when measuring the viscosity of highly viscous materials.

The rotating conicylindrical viscometer has been very useful in studying the behavior of both viscous and non-viscous bitumens under conditions of prolonged shear. Valuable data have been obtained for materials possessing elasticity, thixotropy, and structural viscosity, but systems which have sufficient dispersed phase present to cause dilatancy cannot be successfully tested in a confined viscometer of this type.

FALLING COAXIAL CYLINDER METHOD

The falling coaxial cylinder viscometer, first described by Segel[18] has been used by Pochettino[19] for measuring the viscosity of pitch and by Mack[20] for determining the viscosity of asphaltic materials.

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[18] M. Segel, "A Method for Determining the Internal Friction of Hard Substances," *Physikalische Zeitschrift*, Vol. 4, p. 493 (1903).

The instrument, as used in the past in our laboratories for measuring viscosities above 100,000 poises, and possessing the dimensions of the viscometer used by Pochettino, is shown diagrammatically in Fig. 4. It consists of a hollow brass cylinder *B* having an inner radius equal to 2.592 and a length of 12.69 cm. A solid brass cylinder *C* having a radius of 1.911 cm. and any convenient length is placed inside the hollow one and coaxial with it so as to form an annular space. The two cylinders are heated and placed on an amalgamated brass plate which has pins *D* to center both cylinders. The molten asphalt is then poured into the annular space.

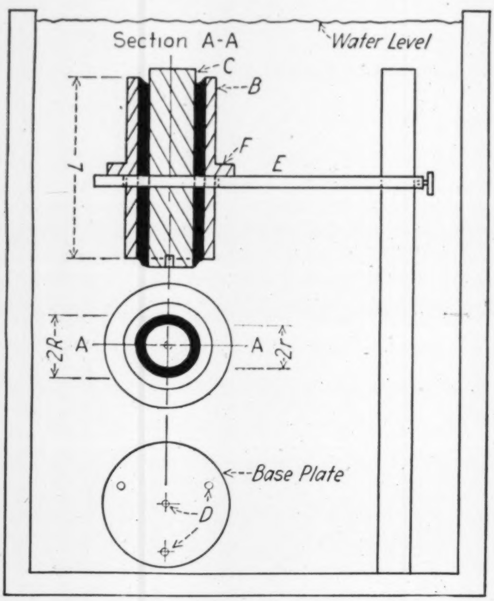

FIG. 4.—Apparatus for Falling Coaxial Cylinder Method.

After the asphalt has cooled, the entire apparatus is placed in a glass-walled constant-temperature water bath for at least 1 hr. before a determination is made. When the asphalt has attained the desired temperature, the base plate is removed and the apparatus placed on a support *E*. It is held in a vertical position by means of the fin *F* and the rate of fall of the inner cylinder is determined by means of a cathetometer. After the inner cylinder has moved out some predetermined distance (usually from 0.3 to 0.5 cm.) the apparatus is reversed and the inner cylinder allowed to return to its original position. A determination usually consists of three "out" and three "in" readings.

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[19] A. Pochettino, "Viscosity and Elasticity of Highly Viscous Materials," *Nuovo cimento*, Vol. 8, p. 77 (1914).

[20] C. J. Mack, "Colloid Chemistry of Asphalts," *Journal of Physical Chemistry*, Vol. 36, p. 2901 (1932).

The equation used for calculating the viscosity is as follows:

$$\eta = \frac{gt}{2\pi Lh}\left[(W - \pi r^2 LD)\log_e \frac{R}{r} + \frac{\pi LD}{2}(R^2 - r^2)\right] \ldots\ldots\ldots(4)$$

where η = viscosity in poises,
g = gravitation constant,
L = length of outer cylinder in centimeters,
h = distance in centimeters of movement in time t (seconds),
R = inner radius of outer cylinder in centimeters,
r = radius of inner cylinder in centimeters,
W = effective weight in grams of applied load (actual weight minus buoyant effect of liquid in bath), and
D = difference in density of material being measured and density of bath in which the apparatus is run in grams per cubic centimeters.

If the bath in which the viscometer is immersed contains a liquid having essentially the same density as the material being tested, the value for the terms containing D in Eq. 4 may be considered as zero and the equation reduces to:

$$\eta = \left(\log_e \frac{\mathrm{R}}{r}\right)\left(\frac{g}{2\pi L}\right)\left(\frac{Wt}{h}\right) = KW\frac{t}{h} \ldots\ldots\ldots\ldots\ldots(5)$$

where K is constant for a particular instrument.

For thixotropic materials the first "out" reading will give an abnormally low rate (high viscosity) but all subsequent readings will approach a constant value. If the material is highly elastic, the initial rate of movement when the direction of flow is reversed will be high. These low and high rates should not be used for calculating the viscosity of the material.

The effect of changing the shearing stress and changing the distance of movement of the inner cylinder has been studied, and it has been found that the mean shearing stress may vary from 220 to 17,000 dynes and the distance of movement from 0.2 to 1.4 cm. without affecting the result within the limits of duplication. However, if the material is quasi-viscous the shearing stress and distance of movement will greatly influence the calculated viscosity and an arbitrary procedure must be set up for determining the apparent viscosity of such materials.

An aluminum inner cylinder may be used to decrease the rate of movement when a soft material is being tested. If, on the other hand, a hard substance is being measured it may be necessary to place an additional weight on the inner cylinder. The rate of movement of the inner cylinder should be regulated for maximum accuracy and convenience. The range of viscosity measured with the falling coaxial cylinder viscometer is limited only by the fastest and slowest rates of movement of the inner cylinder that can be satisfactorily measured.

DEVELOPMENT OF A RAPID METHOD FOR MEASURING HIGH VISCOSITIES

Although the four methods as described above can be used successfully to determine viscosities greater than 1 poise by selecting the method applicable to a particular range, nevertheless their use requires a trained operator and a measurement may take from 1 to 4 hr. Obviously none of them can be used successfully as a control instrument where results are required in the shortest possible time.

When considering the development of a rapid, accurate method the falling coaxial cylinder type of viscometer if modified seemed to possess several possible advantages over the other methods described: namely,

1. The viscometer can be filled and the sample brought to the desired temperature very quickly.
2. It is simple to operate.
3. A small amount of material is required for a test.
4. The method can be used over a wide range of viscosities.

However, the apparatus which had been used and which had essentially the dimensions of the viscometer employed by Pochettino was too large for ease of filling and required too long for a determination. The viscometer was studied thoroughly using instruments of various dimensions. A Venezuelan asphalt (G, in Table II) was measured at 25 C. in twenty-five falling coaxial cylinder viscometers having different dimensions. Since all of the instruments could not be used at the same time, careful attention was given to the heating and aging of the asphalt so that conditions for all measurements would be as nearly the same as possible. The data on asphalt G obtained by means of the various instruments are given in Table I.

The data show that the dimensions of the instrument may have a definite effect on the viscosity value obtained. For example, if the annulus is large with respect to the length of the viscometer, low values generally result. However, if the ratio of the annular width to the length is less than 0.5 the result for instruments of various dimensions agree within the limits of experimental error.

Based on this study an apparatus, which had the following dimensions, was selected for making rapid determinations:

$$R = 1.905 \text{ cm. } (\tfrac{3}{4} \text{ in.})$$
$$r = 1.270 \text{ cm. } (\tfrac{1}{2} \text{ in.})$$
$$L = 2.540 \text{ cm. } (1 \text{ in.})$$
$$\frac{R - r}{L} = 0.25$$

TABLE I.—VISCOSITY OF ASPHALT G USING DIFFERENT FALLING COAXIAL CYLINDER VISCOMETERS.

Viscometer	Dimensions of Viscometer, cm.			$\frac{R-r}{L}$	Viscosity at 25 C., poises × 10^{-6}
	Radius of Outer Cylinder, *R*	Radius of Inner Cylinder, *r*	Length of Asphalt Column, *L*		
No. 1	2.569	0.662	0.520	3.67	1.78
No. 2	2.609	0.662	1.039	1.87	2.30
No. 3	2.569	1.908	0.520	1.27	3.57
No. 4	2.607	0.662	1.590	1.22	3.01
No. 5	3.486	0.662	2.550	1.11	3.32
No. 6	2.607	0.662	1.936	1.01	3.43
No. 7	3.486	1.295	2.550	0.86	3.61
No. 8	2.602	0.662	2.550	0.76	2.53
No. 9	2.609	1.905	1.039	0.68	3.83
No. 10	3.486	1.916	2.550	0.61	3.30
No. 11	2.602	1.295	2.550	0.51	3.72
No. 12	1.912	0.662	2.545	0.49	3.68
No. 13	3.486	2.287	2.550	0.47	3.65
No. 14	2.607	1.916	1.590	0.43	3.74
No. 15	2.607	1.905	1.936	0.36	3.96
No. 16	2.602	1.916	2.550	0.27	4.07
No. 17	1.908	1.270	2.540	0.25	3.95
No. 18	3.486	2.290	5.070	0.24	3.71
No. 19	1.915	1.270	5.053	0.13	4.13
No. 20	2.550	1.906	5.088	0.13	3.88
No. 21	2.592	1.271	12.70	0.10	4.19
No. 22	2.592	1.911	12.69	0.05	4.01
No. 23	2.598	1.943	15.24	0.04	4.20
No. 24	2.591	2.252	12.66	0.03	4.31
No. 25	2.578	1.905	25.40	0.03	4.20

TABLE II.—IDENTIFICATION OF ASPHALTS.

Asphalts A, B, C, F and G were produced from a mixture of heavy gravity with some medium gravity petroleum from the Mene Grande field.

Designation	Source (type)	Process	Penetrations			Softening Point—deg. Fahr., Ring-and-Ball Method
			32 F 200/60	77 F 100/5	115 F 50/5	
A	Venezuelan	Vacuum	13	180	Soft	104
B	Venezuelan	Vacuum	9	138	Soft	108
C	Venezuelan	Vacuum	7	109	Soft	113
D	Californian	Vacuum	7	61	Soft	115
E	Californian	Vacuum	6	55	Soft	117
F	Venezuelan	Vacuum	14	67	Soft	121
G	Venezuelan	Vacuum	12	57	324	124
H	Californian	Vacuum	5	35	244	122
I	Mid-Continent	Vacuum	11	41	229	132
J	Fluxed Refined Trinidad Lake Asphalt	....	13	50	246	129
K	Trinidad	Vacuum	5	18	166	135

TABLE III.—VISCOSITY VALUES OBTAINED FOR ELEVEN ASPHALTS USING SMALL AND LARGE-SIZE FALLING COAXIAL-CYLINDER INSTRUMENTS.

ASPHALT	VISCOSITY, POISES × 10^{-6}	
	SMALL INSTRUMENT	LARGE INSTRUMENT
A	0.320	0.340
B	0.500	0.518
C	0.867	0.879
D	1.91	1.95
E	2.05	2.24
F	2.92	2.74
G	3.95	4.01
H	5.50	5.48
I	9.99	10.6
J	12.2	13.1
K	23.6	23.0

Volume of annular space = 16 cu. cm.

These are essentially the dimensions of instrument No. 17 in Table I, and it should be noted that this instrument gave a viscosity of 3.95×10^6 poises and the average for all instruments with a ratio of annulus to length of less than 0.5 gave an average value of 3.98×10^6 poises.

However, to check this small instrument over a wide range of viscosities, eleven different asphalts were measured simultaneously in it and in the large size viscometer having the dimensions used by Pochettino. The properties of the eleven asphalts are recorded in Table II and the viscosity values obtained using the two viscometers are given in Table III. From the results it appears that the small-size instrument gives about the same viscosity values as are obtained using the large, more cumbersome and less practical viscometer.

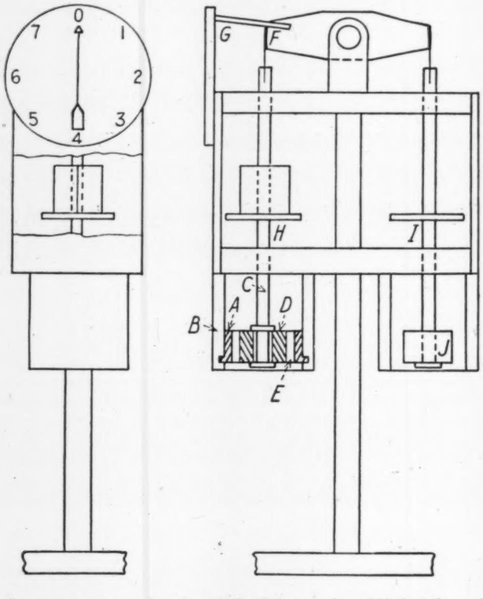

FIG. 5.—Apparatus for Rapidly Measuring High Viscosities.

An apparatus has been developed, using a small instrument of slightly different design, by means of which viscosity can be measured rapidly and accurately by a control operator.

A schematic diagram of the apparatus is shown in Fig. 5. The brass outer cylinder *A* is held in a bakelite holder *B* by means of an arrangement which makes possible rapid insertion or removal of the mold. A bakelite rod *C* passes through the inner cylinder *D*. The material under test fills the annulus *E*. The bakelite rod *C* is connected to a metal ribbon which passes over a rocker arm *F* which in turn is attached to another rod *I* and a counterpoise *J*. A weight *H* applied to rod *C* causes the inner cylinder *D* of the viscometer to move downward; added to rod *I* it causes cylinder *D* to move upward. In order to measure a wide range of viscosities the apparatus is equipped with several weights that may be applied to cause either an upward or a downward movement of the inner cylinder. To protect the weights from corrosion and damage, the entire mechanism is enclosed. The distance of movement of the inner cylinder for a given time is indicated on dial *G*. From the rate of movement under a given load, the viscosity can be calculated directly. A thermostatically controlled bath surrounds the coaxial cylinders and holder *B*. Rod *C* and holder *B* are made of a nonmetallic material in order to eliminate the conduction of heat away from the sample.

The apparatus will measure viscosity in 10 to 100 sec. over a range from 5000 to 1,000,000,000 poises. Further experiments may prove that these limits can be extended. On account of its simplicity and speed of operation this viscometer should be useful to the technologist interested in the flow properties of highly viscous materials such as natural and synthetic resins, tars and asphalts.

DISCUSSION

MR. E. C. BINGHAM[1] (*presented in written form*).—Heretofore it has generally been assumed that materials having the consistency of asphalt are not suitable for viscosity measurements in absolute units. Particularly it has been assumed that the time required would be unjustified by the results obtained. The results obtained by the authors in this and their succeeding papers demonstrate not only that it can be done, but also that the efforts expended are abundantly fruitful, since they prove that many of the samples in common use are to be regarded as true fluids. That discovery simplifies the problems of control.

We are working with the simple property of viscosity. There are perhaps an infinite number of ways of measuring viscosity. There are, no doubt, short methods which can be worked out to save time after these absolute measurements have led the way. It would seem to me possible to relate the quick *relative* measurements to the more deliberate measurements of the research laboratory, so that in the end all of our measurements will be in terms of the same *absolute units*. This will be of immeasurable benefit in that it will make it possible to convert measurements made with one instrument to those made with another having quite different form; and therefore the measurements will be independent of the instruments employed. I would not labor this point were not the whole field of industry cluttered up with a vast array of methods which will appear but a relic of the dark ages as soon as other workers revise or replace the methods now in use.

Freundlich and his co-workers investigated the increase of viscosity of colloidal materials which they called thixotropy. McMillen[2] has attempted to obtain a relation of thixotropy to plastic flow, but in the past no relation has been formed which is sufficiently simple. The present authors have a relation which is easily understandable and which may be used to advantage in studying the behavior of the materials in question.

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[1] Professor of Chemistry, Lafayette College Library, Easton, Pa.

[2] *Journal of Rheology*, Vol. 3, pp. 75, 163 (1932).

THE SUSCEPTIBILITY OF ASPHALTS TO TEMPERATURE CHANGE

By H. E. Schweyer,[1] C. E. Coombs[2] and R. N. Traxler[2]

Synopsis

The viscosity-temperature relationships of asphaltic materials are complex and no type of equation has been found that is of practical value over a wide temperature range. However, at processing temperatures where the asphalts have viscosities ranging from 1 to 5 poises and at service temperatures (15 to 35 C., 59 to 95 F.) a log-viscosity *versus* temperature plot is a straight line within the limits of experimental error. For these regions a new, simple and logical way of expressing susceptibility based on the percentage change of viscosity for a 1 deg. Cent. rise in temperature is proposed, and is called the Asphalt Viscosity Index.

Using the Saybolt Furol viscosimeter for measuring consistencies between 1 and 5 poises (approximately 50 to 250 Furol seconds) and the falling coaxial cylinder viscometer for evaluating viscosities at service temperatures (15 to 35 C., 59 to 95 F.) the susceptibility of an asphalt in terms of absolute units can be determined very quickly.

Viscosity data in absolute units are given for a number of different paving asphalts both at the high "processing" and the low "service" temperatures. The Asphalt Viscosity Indices for each asphalt at both temperature ranges are recorded together with susceptibility factors for some of the asphalts calculated from penetration data. The indices of the various asphalts are greatly different in magnitude for the two temperature ranges and the relative order of susceptibility may change.

Data are given which indicate that the susceptibility of a viscous bitumen-filler mixture to temperature change is very nearly the same as that of the bitumen present.

The methods commonly used for evaluating the susceptibility of a bituminous material, that is, the change of consistency with change in temperature, are dependent on arbitrary tests such as penetration, softening point, or time of efflux at an elevated temperature. Although consistency as measured by all of these empirical tests is some function of viscosity expressed in terms of absolute units, the relationship is frequently complicated as has been shown for penetration.[3] Since viscosity is a fundamental property of all fluid material, the changes in consistency brought about by changes in temperature should be most satisfactorily expressed in terms of absolute viscosity units.

[1,2] Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.

[3] R. N. Traxler, C. U. Pittman and F. B. Burns, "Rheological Properties of Asphalt II. Discussion of Penetration—Viscosity Relationships," *Physics*, Vol. 6, p. 58 (1935).

However, the viscosity-temperature relationships of asphaltic materials are complex and no type of equation has been found that is of any practical use over a wide temperature range. A plot of the logarithm of the viscosity *versus* the temperature shows a rapid change in viscosity over a wide temperature range, but at low and high temperatures the log viscosity - temperature plot is a straight line within the limits of experimental error. Therefore, for these regions

$$\log \eta = a\,t + \log b \qquad \text{(1)}$$

where η = viscosity in poises,
t = temperature in degrees Centigrade, and
a and b are constants.

For paving asphalts this equation holds between 15 and 35 C. (59 and 95 F.), a temperature range to which the asphalts are frequently exposed in service. Also, with different constants, the same type of equation applies for asphalts over a viscosity range from 1 to 5 poises. This is the consistency range at which asphalts are sufficiently fluid to work easily in a paving plant. Thus, fortunately, Eq. 1 satisfactorily expresses the viscosity-temperature relationship at the service temperatures and also at the temperatures at which the asphalts are handled.

Traxler and Schweyer[4] have reviewed and discussed the various arbitrary methods that have been proposed for measuring the susceptibility of bitumen. A method frequently used is:

$$\text{Susceptibility factor}^{5} = \text{Penetration at 41.6 C. (115 F.), 50-g. weight, 5 sec.} - \left\{\frac{\text{Penetration at 0 C. (32 F.), 200-g. weight, 60 sec.}}{\text{Penetration at 25 C. (77 F.), 100-g. weight, 5 sec.}}\right\} \qquad \text{(2)}$$

Another relationship sometimes used is

$$\text{Susceptibility factor} = \frac{\text{Penetration at 38 C. (100 F.), 100-g. weight, 5 sec.}}{\text{Penetration at 25 C. (77 F.), 100-g. weight, 5 sec.}} \qquad \text{(3)}$$

Both Eqs. 2 and 3 may be criticized mainly because they measure susceptibility under unknown and changing stresses which change at different rates.

Holmes, Collins and Child[6] recently established three indices for evaluating the susceptibility of asphalt to temperature change. At atmospheric temperatures they used a relationship between penetration at 25 C.

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[4] R. N. Traxler and H. E. Schweyer, "Rheological Properties of Asphalts III. A Viscosity Index," *Physics*, Vol. 7, p. 67 (1936).

[5] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1933 Book of A.S.T.M. Standards, Part II, p. 971.

[6] A. Holmes, J. O. Collins and W. C. Child, "Measuring the Susceptibility of Asphalts to Temperature Change," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 8, p. 100 (1936).

(77 F.), 100-g. weight, 5 sec. and the ring-and-ball softening point. The ratio between the penetration at 25 C. (77 F.), 100-g. weight, 5 sec. and that at 0 C. (32 F.), 200-g. weight, 60 sec. was used for low temperatures. To measure the susceptibility at high temperatures they suggested a relationship between Furol viscosity at 135 C. (275 F.) and penetration at 25 C. (77 F.), 100-g. weight, 5 sec.

Since then Nevitt and Krchma[7] have proposed an index called the viscosity-temperature susceptibility coefficient (V.T.S.) which is based on the A.S.T.M. viscosity-temperature chart[8] for liquid petroleum products.

THE ASPHALT VISCOSITY INDEX (A.V.I.)

If, as has been proposed elsewhere,[9] the change of *viscosity* with temperature is accepted as a practical and logical definition of susceptibility it follows that the susceptibility will be a function of some viscosity-temperature relationship like that given by Eq. 1. Where such a relationship exists the percentage change of viscosity is given by:

$$\text{Percentage change} = 100\left(\frac{\eta_2 - \eta_1}{\eta_1}\right) = 100\left(\frac{\eta_2}{\eta_1} - 1\right) \ldots\ldots (4)$$

where η_1 and η_2 are the viscosities at temperatures t_1 and t_2, respectively.

Percentage change is a function of the slope a of Eq. 1 because

$$a = \frac{\log \eta_2 - \log \eta_1}{t_2 - t_1} = \frac{\log(\eta_2/\eta_1)}{t_2 - t_1} \ldots\ldots\ldots\ldots (5)$$

therefore, $\log \eta_2/\eta_1 = a\,(t_2 - t_1) \ldots\ldots\ldots\ldots (6)$

and $\eta_2/\eta_1 = 10^{a(t_2 - t_1)} \ldots\ldots\ldots\ldots (7)$

Consequently, Eq. 4 becomes

Percentage change $= 100\,(10^{a(t_2 - t_1)} - 1) \ldots\ldots\ldots\ldots (8)$

which states that the percentage change in viscosity for a given interval of temperature is constant over any temperature range where Eq. 1 applies. The percentage change in viscosity of asphalts for a particular interval of temperature rise is smaller than for an equal interval of temperature drop.

The susceptibility of an asphalt may then be measured in terms of a viscosity index which is the percentage decrease in viscosity for a 1 deg. Cent. rise in temperature. The numerical value of the index is therefore given by Eq. 8 where $t_2 - t_1$ is equal to 1. Experimentally, it is only necessary to measure the viscosities η_a and η_b at any two temperatures t_a

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[7] H. G. Nevitt and L. C. Krchma, "The Viscosity Temperature Susceptibility Coefficient as an Index of the Effect of Temperature on the Consistency of Asphalts," Presented before the Petroleum Division, Am. Chemical Soc., Kansas City, Mo., April 13-17, (1936).

[8] Tentative Standard Viscosity Temperature Chart for Liquid Petroleum Products (D 341 - 32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 772 (1932); also 1935 Book of A.S.T.M. Tentative Standards, p. 775.

[9] R. N. Traxler and H. E. Schweyer, "Rheological Properties of Asphalts III. A Viscosity Index," *Physics*, Vol. 7, p. 67 (1936).

and t_b (deg. Cent.) respectively, within a temperature range where Eq. 1 is valid. Then the Asphalt Viscosity Index[10] may be expressed as:

$$\text{A.V.I.} = 100\,(10^a - 1) = 100\,[(\eta_a/\eta_b)^{1/(t_a - t_b)} - 1] \ldots\ldots (9)$$

Since the viscosity of asphalt decreases with temperature the values of the A.V.I. are negative, but from the definition given above the sign may be ignored. It should be noted that the A.V.I. has a practical meaning because it evaluates the susceptibility of the asphalt in terms of percentage change of viscosity (in poises) for 1 deg. Cent. rise in temperature.

Asphalts Studied:

In Table I a record is given of the sources, methods of processing and identifying properties of the fourteen asphalts of paving consistency which were studied.

MEASUREMENT OF VISCOSITIES

Measurements of the viscosities of the asphalts at temperatures ranging from 15 to 35 C. (59 to 95 F.) were made by means of the alternating stress method[11,12,13] and are given in Table II. This method is rather time-consuming and the same information could have been obtained more rapidly by means of the falling coaxial cylinder viscometer.[13]

Determinations at high temperatures where the viscosities of the asphalts varied from one to five poises were made by means of the Saybolt Furol viscosimeter.[14] For each asphalt four or five measurements were made with this instrument over the range 50 to 250 Furol seconds. By means of the formula[15]

$$\frac{\eta}{d} = 0.0216\,F \ldots\ldots\ldots\ldots\ldots\ldots\ldots (10)$$

where η = viscosity in poises,
d = density in grams per cubic centimeter, and
F = Furol seconds.

the Furol viscosities were converted to absolute units (poises). From the data given in Table I the densities of the asphalts at the higher temperatures were calculated, using the formula given by Cragoe.[16] The logarithms of the viscosity values in poises were plotted against the temperature at which the determinations were made. Because a thermostatically controlled Furol viscosimeter was not used, the experimental temperatures were not easily established at definite regular intervals. In order to obtain data at 5 deg. Cent. (9 deg. Fahr.) intervals, as was done at the low temperatures, values were selected from a semi-log plot of the experimental data by

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[10] Actually the A.V.I. may be calculated from the expression, 100 (anti-logarithm of the slope $a - 1$).

[11] E. C. Bingham and R. A. Stephens, "The Alternating Stress Method for the Measurement of the Consistency of Very Stiff Materials," *Physics*, Vol. 5, p. 217 (1934).

[12] C. U. Pittman and R. N. Traxler, "Rheological Properties of Asphalts. I. Effect of Temperature," *Physics*, Vol. 5, p. 221 (1934).

[13] R. N. Traxler and H. E. Schweyer, "Measurement of High Viscosity—A Rapid Method," see p. 518.

[14] Tentative Method of Test for Viscosity of Petroleum Products and Lubricants (D 88 – 33), 1933 Book of A.S.T.M. Standards, Part II, p. 880.

[15] International Critical Tables, Vol. 1, pp. 32–33, McGraw-Hill Book Co., Inc., New York City (1926).

[16] C. S. Cragoe, "Thermal Properties of Petroleum Products," National Bureau of Standards *Miscellaneous Publication No. 97* (1929).

Table I.—Identification of Asphalts.

Designation	Source (type)	Process	Penetrations				Softening point, Ring-and-Ball Method, deg. Fahr.	Density 15.56 C. (60 F.), g. per cu. cm.
			32 F., 200-g. weight, 60 sec.	77 F., 100-g. weight, 5 sec.	100 F., 100-g. weight, 5 sec.	115 F., 50-g. weight, 5 sec.		
A	Mexican	Batch steam	17	62	...	...	126	1.050
B	Venezuelan		18	61	...	282	126	1.041
C	Trinidad		11	60	246	295	118	1.034
D	Fluxed refined Bermudez Lake asphalt	....	16	61	216	298	122	1.061
E	Fluxed refined Trinidad Lake asphalt	....	16	62	204	300	125	1.220
F	Californian	Vacuum	5	35	169	244	122	1.023
G	Californian		6	55	..	soft	117	1.021
H	Mid-Continent		11	41	148	229	132	1.032
I	Mid-Continent		13	56	193	305	124	1.027
J	Mid-Continent		15	63	...	soft	123	1.025
K	Venezuelan		9	34	120	175	133	1.034
L	Venezuelan		12	57	196	324	124	1.028
M	Venezuelan	Batch steam	16	57	177	265	124	1.040
N	Mexican	Vacuum	17	55	150	272	126	1.048

Table II.—Viscosity-Temperature Data.

Temperature		Asphalt A	Asphalt B	Asphalt C	Asphalt D	Asphalt E	Asphalt F	Asphalt G	Asphalt H	Asphalt I	Asphalt J	Asphalt K	Asphalt L	Asphalt M	Asphalt N
deg. Cent.	deg. Fahr.														
		Viscosity, poises $\times 10^{-6}$													
15	59	92.0	63.3	64.9	36.9	43.2	....	....	....	....	....	....	....	....	....
16	61	....	....	....	....	....	....	43.6	....	....	....	....	....	....	....
20	68	22.6	17.6	14.7	....	12.5	32.5	....	....	....	....	50.3	19.6	16.5	20.3
21	70	....	....	....	9.57	9.49	....	11.4	40.2	14.1	11.0	....	....	....	....
25	77	6.98	6.24	3.35	3.66	4.06	9.19	3.22	13.6	4.91	3.89	14.0	4.58	5.47	6.99
30	86	2.75	1.81	0.902	1.28	1.32	2.13	0.849	4.11	1.45	1.28	4.31	1.60	2.15	2.35
35	95	0.845	0.497	0.250	0.491	0.484	0.674	....	1.18	0.508	0.417	1.37	0.517	0.610	0.727
		Viscosity, poises													
125	257	....	....	....	....	....	....	4.49	....	....	....	....	....	....	....
130	266	....	....	4.05	....	....	4.46	3.39	....	....	....	....	....	....	....
135	275	....	4.80	3.00	....	....	3.48	2.60	....	....	....	....	....	4.31	....
140	284	....	3.55	2.25	3.78	....	2.72	1.99	....	4.78	4.36	5.53	4.09	3.39	....
145	293	4.20	2.68	1.71	2.88	....	2.16	1.55	4.11	3.84	3.43	4.32	3.21	2.63	....
150	302	3.19	2.10	1.33	2.22	....	1.71	1.21	3.44	3.12	2.87	3.41	2.53	2.15	4.22
155	311	2.42	1.64	1.05	1.73	....	1.38	....	2.84	2.52	2.31	2.70	2.01	1.71	3.40
160	320	1.92	1.33	....	1.39	....	1.11	....	2.36	2.07	1.94	2.16	1.60	1.39	2.73
165	329	1.57	1.11	....	1.13	....	....	....	1.97	1.70	1.59	1.75	1.29	1.14	2.22
170	338	1.28	....	....	....	....	....	....	1.64	1.43	1.35	1.42	1.07	....	1.81
175	347	1.08	....	....	....	....	....	....	1.37	1.18	1.12	1.15	....	....	1.49
180	356	....	....	....	....	....	....	....	1.17	....	..	...	....	....	1.22
185	365	....	....	....	....	....	....	....	....	....	....	....	....	....	1.02

interpolation and extrapolation. The viscosity data are recorded in Table II. In cases where sufficient experimental data are spread uniformly over the range one to five poises they may be used directly to calculate the susceptibility of the asphalt.

The Asphalt Viscosity Indices were calculated from the data in Table II for each of the asphalts for the two temperature ranges and the values are recorded in Table III. For comparison the susceptibility factors calculated by Eqs. 2 and 3 are included in Table III for those asphalts on which data could be obtained.

TABLE III.—SUSCEPTIBILITY OF VARIOUS ASPHALTS.

Asphalt	Type	A.V.I. (15 to 35 C., 59 to 95 F.)	A.V.I. (1 to 5 poises)	Susceptibility Factors	
				Eq. 2	Eq. 3
M...........	Venezuelan	19.0	4.4	4.4	3.1
N...........	Mexican	20.0	4.0	4.6	2.7
D...........	Bermudez	20.0	4.8	4.6	3.5
E...........	Trinidad Lake	20.0	...	4.6	3.3
J...........	Mid-Continent	20.5	3.8	...	...
A...........	Mexican	20.5	4.4	...	...
L...........	Venezuelan	21.0	4.4	5.5	3.4
B...........	Venezuelan	21.0	4.8	4.3	...
I...........	Mid-Continent	21.0	4.0	5.2	3.4
K...........	Venezuelan	21.5	4.4	4.9	3.5
H...........	Mid-Continent	21.5	3.6	5.3	3.6
F...........	Californian	23.5	4.6	6.8	4.8
C...........	Trinidad oil	24.5	5.2	4.7	4.1
G...........	Californian	24.5	5.2	...	...

TABLE IV.—VISCOSITY-TEMPERATURE DATA FOR BITUMENS AND MIXTURES WITH VARIOUS MINERAL POWDERS.

Volume, per cent		Viscosity, poises × 10^{-6}					A.V.I.
		16 C.	21 C.	25 C.	30 C.	35 C.	
Asphalt G	*Mineral*						
100	0	43.6	11.4	3.22	0.849		24.5
90	10 (red slate)	67.2	14.9	5.29	1.27		24.5
90	10 (silica)	61.8	14.0	4.46	1.24		24.5
80	20 (trap rock)	104	22.2	7.35	2.01		24.5
Asphalt H	*Limestone*						
100	0		40.2	13.6	4.11	1.18	21.5
84.2	15.8		62.0	21.0	6.91	2.15	20.0
72.7	27.3		98.3	35.7	11.2	3.20	20.5
64.0	36.0		137	41.4	13.6	4.05	20.5
Asphalt I	*Limestone*						
100	0		14.1	4.91	1.45	0.508	21.0
84.2	15.8		26.7	9.90	3.18	1.06	20.5
64.0	36.0		59.6	22.5	7.34	2.46	20.5

FILLED BITUMENS

Since in practice most bitumens are used in combination with pulverized solids, which serve as fillers or stabilizing agents, the important question arises whether the addition of mineral powder to a bitumen changes the viscosity-temperature relationships. If we are to apply the viscosity index discussed above, the investigation must be confined to bitumen - mineral powder mixtures which are viscous, and this requirement immediately places a limitation upon the amount of filler which may be employed. Asphalt G of Table I was mixed with different mineral powders and the viscosities of the resulting mortars were measured at temperatures from 16 to 30 C. (61 to 86 F.) by means of the alternating stress method.[17] Asphalts H and I were also mixed with different amounts of limestone dust. Table IV gives the composition of and viscosity data on the various mixtures. The data for the Californian asphalt and its mortars are shown graphically in Fig. 1. It appears that a viscous mixture of a bitumen and mineral filler has an A.V.I. only slightly lower than that of the bitumen present.

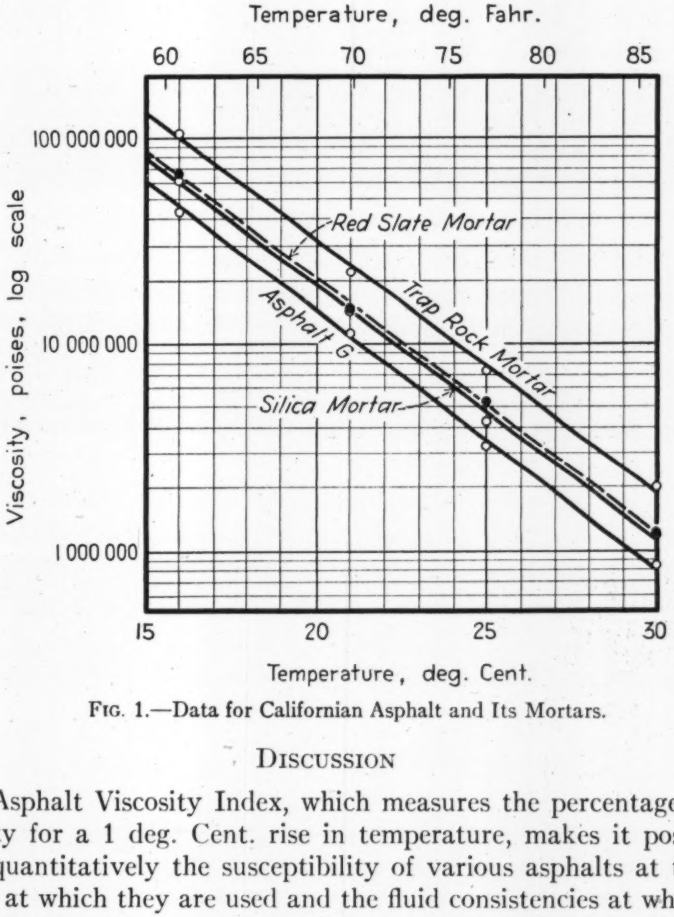

FIG. 1.—Data for Californian Asphalt and Its Mortars.

DISCUSSION

The Asphalt Viscosity Index, which measures the percentage change of viscosity for a 1 deg. Cent. rise in temperature, makes it possible to compare quantitatively the susceptibility of various asphalts at the high viscosities at which they are used and the fluid consistencies at which they are handled. For these two temperature ranges the indices of each asphalt are greatly different in magnitude. Further, the relative order of susceptibility for the various asphalts may be different at the two ranges.

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[17] E. C. Bingham and R. A. Stephens, "The Alternating Stress Method for the Measurement of the Consistency of Very Stiff Materials," *Physics*, Vol. 5, p. 217 (1934).

When a mineral powder is added to a bitumen the viscosity of the resulting mixture is much greater than that of the bitumen present. This change in consistency sometimes caused the assumption to be made that the addition of a filler caused a considerable decrease in the susceptibility to temperature change. However, measurements of the effect of temperature in terms of percentage change of viscosity for a one degree rise in temperature have indicated that the susceptibility of a bituminous mortar is practically the same as that of the bitumen present.

By using the Saybolt Furol viscosimeter for measuring viscosities at the high temperatures used in an asphalt paving plant (consistencies between 50 and 250 Furol seconds) the susceptibility of a bitumen may be determined very quickly. For the service temperatures (below 35 C., 95 F.) the alternating stress method has been used but a modified form of the falling coaxial cylinder viscometer is recommended because of the greater rapidity with which measurements can be made.

Acknowledgment.—The authors are indebted to Mr. L. R. Moffatt for assistance in the experimental work.

DISCUSSION

Mr. R. R. Thurston[1] (*presented in written form*).—The authors of this paper are to be congratulated upon the thoroughly scientific manner in which they have approached a question over which there has been considerable controversy and lack of fundamental data. It is interesting to note that the maximum variation in viscosity indices between different paving asphalts determined at temperatures below 35 C. (95 F.) is approximately 20 per cent, whereas the variation when determined on the liquid material is approximately 35 per cent. All indices are much higher at the low-temperature range than at the high-temperature range.

Differences between the susceptibility of asphalts used in paving mixtures are frequently evidenced by the behavior of pavements at high summer temperatures. These temperatures frequently exceed the melting point of these products, and 60 C. (140 F.) is not unusual. Viscosity data at temperatures in the range of 50 to 60 C. (120 to 140 F.) would, therefore, be of considerable interest in this connection.

As pointed out in the paper, the log viscosity-temperature curve is probably not a straight line in this temperature range. The behavior of asphalts at these temperatures cannot be predicted from the data which are presented at temperatures below 35 C. (95 F.) and at high temperatures at which the materials have a Furol viscosity of from 50 to 250 sec.

It would be interesting to continue the work reported by the authors, including data on viscosities at intermediate temperatures in the range of 40 to 65 C. (105 to 150 F.)

Mr. R. N. Traxler.[2] We have data on a number of asphalts over the temperature range from 15 to 130 C. a portion of which has been published.[3] From these data it is possible to calculate the ratio between the viscosities at 60 C. (140 F.) and 25 C. (77 F.), but for estimating the susceptibility of the asphalt at 60 C. (140 F.) or over the range 25 to 60 C. this ratio is as meaningless as some of the other so-called susceptibility factors which appear in the literature. However, the A.V.I. may be obtained in the neighborhood of 60 C. (140 F.) with sufficient accuracy to warrant its use. It is only necessary to determine the slope of the log viscosity-temperature curve in the region of 60 C. (140 F.). Graphical methods are not necessary; an average slope can be determined analytically (Eq. 6) from the viscosities of each asphalt at 55 C. (131 F.), 60 C. (140 F.) and 65 C. (149 F.) and the A.V.I. values derived therefrom. By considering only this narrow temperature range the A.V.I. is sufficiently accurate (expressed to the nearest 0.5 unit) and represents the percentage decrease in viscosity for a 1 deg. Cent. rise in temperature at 60 C. (140 F.).

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[1] Chemical Engineer, Technical Division, The Texas Company, New York City.

[2] Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.

[3] C. U. Pittman and R. N. Traxler, "Rheological Properties of Asphalt I. Effect of Temperature," *Physics*, Vol. 5, p. 221 (1934).

The following table gives the values for the *average* A.V.I. of the various asphalts at 60 C. (140 F.):

ASPHALT	TYPE	A.V.I 55 to 65 C. (131 to 149 F.)
M	Venezuelan	12.5
N	Mexican	12.0
D	Bermudez	14.0
E	Trinidad Lake	12.5
J	Mid-Continent	13.0
A	Mexican	13.5
L	Venezuelan	14.0
B	Venezuelan	14.0
I	Mid-Continent	12.0
K	Venezuelan	13.5
H	Mid-Continent	13.5
F	Californian	13.0
C	Trinidad Oil	12.5
G	Californian	13.5

Comparison with Table III of the paper shows that the order of the asphalts is different at 60 C. (140 F.) than at either the service or processing temperatures.

MR. E. C. BINGHAM[4] (*presented in written form*).—At the World Petroleum Congress in London, R.N.J. Saal presented a paper on "Determinations Regarding the Plastic Properties of Asphaltic Bitumens."[5] He has also used:

1. The capillary instrument in two forms (*a*) the Redwood, and (*b*) the Vogel Ossag viscometer;
2. The sinker viscometer;
3. The pressure capillary viscometer;
4. The Couette Concentric Cylinder viscometer;
5. The Pochettino viscometer; and
6. The rod viscometer.

He found the following viscosity-temperature relation to hold for nonplastic bitumens.

$$\log \log (V_k + 0.95) = -m \log T + C$$

where V_k is the kinematic viscosity in centistokes.

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[4] Professor of Chemistry, Lafayette College Library, Easton, Pa.

[5] *Proceedings*, World Petroleum Congress (London), Vol. II, p. 515 (1933); *Journal*, Inst. Petroleum Technologists (London), Vol. 19, p. 176 (1933).

He studied the relation between viscosity and penetration and gives it as:

$$\log_{10} = -1.93 \log_{10} \text{penetration} + 9.71$$

He also reports on several plastic asphaltic bitumens.

The plasticity is due to asphaltenes which may be removed and the asphalt is then found to be no longer plastic.

Saal found the alteration in viscosity of asphaltic bitumens with the temperature to be wholly or almost wholly due to expansion. A rise in temperature of 1 deg. Cent. might be compensated for by an increase of pressure of 18 atmospheres.

Saal found that soft asphaltic bitumens do not show thixotropy to a marked degree but hard ones show the property very strongly. A yield value was indicated for the hard asphaltic bitumens. Also elasticity was marked in the plastic types.

In offering a simple device for getting the viscosities of hard materials quickly in absolute units and comparing them, the authors have made possible a marked advance.

MR. C. L. MCKESSON[6] (*presented in written form*).—This carefully prepared paper presents a new way of expressing changes in consistency produced by changes in temperature—sometimes referred to as "susceptibility"—and from an academic viewpoint is interesting and ingenious. It is not to be inferred, however, that the so-called "susceptibility" may be considered as an index or an indication of the suitability of an asphalt for use in pavements, but merely one of many proposed "identification tests" by which asphalts from certain sources may be excluded or specified without actually naming the source. Even as such a test, it may easily fail of its objective because asphalt, from almost any source, may, by variations in the process of refining, be made to show "susceptibility" ratios not found in the same asphalt refined by the usual methods. If identification tests are desired, there are many more positive than the so-called "susceptibility."

If it was intended to infer that A.V.I. is an important property and that a higher "susceptibility factor" indicates unsuitable properties in the asphalt, it was unfortunate that an asphalt was singled out in Fig. 1 which has a service record of successful use over 40 yr. under every climatic condition encountered in the United States, including regions where summer temperatures of 120 F. are frequently encountered and others where winter temperatures of −40 F. are not unusual. Pavements built with California asphalt, presumably those referred to in Fig. 1, have excellent service records. If, as the paper suggests, "susceptibility factors" are high for California asphalts, then this splendid service record completely refutes any idea that the "susceptibility factor" bears any relationship to actual pavement performance.

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[6] Director of Engineering and Research, American Bitumuls Co., San Francisco, Calif.

Stability in asphalt pavements is not secured by using an asphalt which will not soften when heated or harden when cold, but by the proper gradation of proper aggregates and by properly controlling the asphaltic content.

In Table IV data are shown which are said to prove that the A. V. I. of certain asphalts does not change with the addition of various (inert) mineral powders. This is probably true of all asphalts when inert mineral fillers are added to the liquid asphalt in small quantities. Fillers are not used to change the asphalt but to impart stability to paving mixtures to which the fillers are added. The use of an excessive quantity of asphalt partially or wholly nullifies the stabilizing effect of the filler as shown by Hubbard in his paper on "Fillers" presented to the Association of Asphalt Paving Technologists in 1927. In the example cited in Table IV, the percentage of mineral filler ranged from 13 per cent to 36 per cent combined with 90 to 64 per cent of asphalt. Obviously the only effect would be to suspend the filler in the asphalt and thereby secure a higher viscosity at all temperatures than the viscosity of the pure asphalt.

MR. J. S. MILLER, JR.[7]—As one who has assisted in directing the work presented in this paper, I want to say that it was not undertaken with any idea of using it from the commercial standpoint. The data are presented as determined and we have been frank enough to identify the asphalts used. Many papers upon viscosity of these highly viscous materials make no mention of the character or source of the asphalt used, which always places one at a great disadvantage when trying to make comparisons of results.

Reference to the paper on "Increase in Viscosity of Asphalts with Time,"[8] will show California asphalt in a rather enviable position in that it shows the least time hardening of any asphalt investigated. Back of this piece of work and the time hardening is the broader question of the actual structure of asphalt and the effect of fillers on that structure. That is what we are aiming at, to find out what that effect is, because fillers vary with respect to the permanency of properties and performance of an asphalt in which they are incorporated, particularly as to weather exposure. The disclosed work was not carried far enough to show that fillers of different chemical composition behave differently. If you try the pigment type such as zinc oxide and white lead you find a definite and marked effect which may be chemical, but as yet we do not know.

We are studying the physical effect of fillers upon asphalt, and these are some of the yardsticks we are developing to measure these differences.

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[7] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

[8] See p. 544.

Fillers are added in a paving mixture to give us physical modification and stabilization of the asphalt.

MR. TRAXLER.—I want to emphasize Mr. Miller's statement in regard to the desirability of including identification of the material under investigation. We have always argued that any published information concerning asphalt should state the origin, method of processing and give identifying characteristics. Although its susceptibility to temperature change affects the applicability of an asphalt for certain uses, we are not attempting to set up the A. V. I. as a "quality" test for bituminous materials. However, this index does measure and evaluate one of the important properties of any bituminous material.

INCREASE IN VISCOSITY OF ASPHALTS WITH TIME

By R. N. Traxler[1] and H. E. Schweyer[2]

Synopsis

When asphalts were maintained undisturbed at 25 C. (77 F.) the viscosities were found to increase with time. This aging phenomenon has been studied by means of the falling coaxial cylinder viscometer for a number of asphalts of various origins and methods of processing.

Eight to eleven viscometers were loaded with the asphalt to be investigated and placed in a cabinet maintained at 25 C. (77 F.). As soon as the samples had cooled to this temperature (3 to 4 hr.) one instrument was removed and the viscosity determined at 25 C. (77 F.). The remaining samples were removed at increasing intervals of time and the viscosities measured.

From the data obtained it is evident that certain asphalts age harden more rapidly and extensively than others, and that the phenomenon in air-blown asphalt is different than in steam- or vacuum-refined materials. Adding mineral powder to an asphalt has very little effect on the rate of age hardening. An Asphalt Aging Index is proposed which quantitatively evaluates the rate of increase of viscosity with time. Development of a thermally unstable structure within the asphalt is probably the cause of the age-hardening phenomenon.

Reheating a time-hardened sample *in situ* returned the asphalt to its original viscosity provided too long a time had not elapsed. However, if a sample was aged for several thousand hours, permanent changes (due to volatilization, etc.) also occurred in the asphalt and reheating the sample did not eliminate all of the viscosity developed during aging.

It is recognized by bituminous technologists that the penetration test for consistency may give low values if 2 or 3 days are permitted to elapse between pouring the sample and making the determination. A number of preliminary studies concerning this phenomenon of "time-hardening" or aging, extending over several weeks, indicated that the hardening was not caused by surface evaporation since both open and tightly closed containers showed about the same degree of hardening. Further, gentle reheating of the samples brought their penetrations back almost to the original values. Although the existence of the phenomenon of age-hardening was established from penetration data it developed that this method of determining consistency gave erratic results over an extended period of time and it became apparent that a more sensitive method of measurement was needed. Soft-

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[1,2] Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.

ening point determinations were tried and discarded because the heating destroyed the increased consistency developed over a period of time.

Viscosity determined in absolute units at atmospheric temperatures seemed to offer a simple and sensitive method of measuring this aging phenomenon, provided a viscometer were available in which the sample could be allowed to age *in situ* over an extended period of time. Such an instrument was found in the falling coaxial cylinder type of viscometer as proposed by Segel[3]; used by Pochettino,[4] and recently described and discussed by Traxler and Schweyer.[5]

ASPHALTS

The asphalts studied were, with one exception, steam- and vacuum-refined asphalts of paving consistency. Table I gives the identifying properties of the ten asphalts investigated.

Table I.—Identification of Asphalts.

Designation	Source	Process	Penetration at 25C. (77 F.) 100-g. weight, 5 sec.	Softening Point, deg. Fahr., Ring-and-Ball Method
A...........	Californian	Vacuum	55	117
B...........	Trinidad	Batch steam	55	121
C...........	Fluxed Refined Trinidad Lake Asphalt	....	56	127
D...........	Venezuelan	Vacuum	57	124
E...........	Mid-Continent	Vacuum	56	123
F...........	Venezuelan	Batch steam	63	125
G...........	Californian	Vacuum	35	122
H...........	Mexican	Vacuum	55	126
I...........	Venezuelan	Batch steam	53	128
J...........	Venezuelan	Air-blown	95	132

EXPERIMENTAL PROCEDURE AND RESULTS

Eight to eleven falling coaxial cylinder viscometers were filled simultaneously with each of the above asphalts. These viscometers were made up of a hollow brass outer cylinder 12.69 cm. long with an inside radius of 2.592 cm. and a solid brass inner cylinder (longer than the outer one) having a radius of 1.911 cm. The cylinders were heated in an oven to 110 C. (230 F.) in order to facilitate filling the annular space between them with hot asphalt. Then, the two cylinders were placed on an amalgamated base plate equipped with pins which centered them. After the viscometers and contents had cooled to room temperature they were trimmed of any extraneous asphalt and placed in a constant temperature air bath at 25 C.

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[3] M. Segel, "A Method for the Determination of the Inner Friction of Solid Materials," *Physikalische Zeitschrift*, Vol. 4, p. 493 (1903).

[4] A. Pochettino, "On Properties of Plastic Bodies," *Nuovo cimento*, Vol. 8, p. 77 (1914).

[5] R. N. Traxler and H. E. Schweyer, "Measurement of High Viscosity—A Rapid Method," see p. 518.

±0.5 deg. Cent. (77 F. ±1 deg. Fahr.). At increasing intervals of time a loaded viscometer was removed from the bath and the viscosity of the asphalt determined at 25 C. (77 F.). Thus, values were obtained on samples of a given asphalt which had aged at a constant temperature for varying lengths of time. In these experiments all of the asphalts were run under approximately the same mean shearing stress (7200 dynes per sq. cm.) and the distance of movement of the inner cylinder was 0.58 cm.

After the viscosities of asphalts A, E, H, and J were determined for the last instruments, the viscometers and contents were heated by means of a gas flame to a temperature above the softening point of the asphalts. After cooling, the viscosities were determined at 25 C. (77 F.) as recorded in parenthesis in Table II. It will be noted that reheating asphalts, which had time-hardened for several thousand hours, destroyed a large percentage of the viscosity which had developed during aging. A sample of asphalt I after age hardening for 640 hr. was remelted *in situ* and cooled. The viscosity at 25 C. (77 F.) was found to be slightly less than the original value because the viscosity of the remelted sample was determined after 3 hr. whereas the original measurement was made after 4 hr. had elapsed. Thus, if too much time has not elapsed, the aging effect may be eliminated by simply remelting the sample. However, if the material has been standing in the viscometer for several thousand hours a permanent hardening (due to evaporation or other changes in the asphalt) may occur simultaneously

TABLE II.—VISCOSITY DATA FOR THE HARDENING OF ASPHALT WITH TIME AT 25 C. (77 F.).

Time of Aging, hr.	Viscosity, poises (× 10^{-6})	Time of Aging, hr.	Viscosity, poises (× 10^{-6})	Time of Aging, hr.	Viscosity, poises (× 10^{-6})	Time of Aging, hr.	Viscosity, poises (× 10^{-6})	Time of Aging, hr.	Viscosity, poises (× 10^{-6})
Asphalt A		Asphalt B		Asphalt C		Asphalt D		Asphalt E	
3	2.11	3.5	3.06	3.5	3.44	3.5	3.76	4.5	3.77
24	2.12	22	3.18	25	3.65	25	3.95	24	4.23
51	2.14	46	3.19	52	3.86	51	4.18	72	4.47
96	2.14	214	3.38	172	3.92	100	4.23	144	4.27
168	2.18	410	3.43	508	4.06	172	4.20	243	4.49
336	2.25	430	3.41	908	4.25	268	4.41	411	4.56
936	2.26	599	3.52	2212	4.40	676	4.42	649	4.70
2523	2.32	1031	3.53			1036	4.58	1371	4.79
6048	2.43	3362	3.58			2040	4.64	3003	5.02
8834	2.49					7251	5.20	5150	5.27
(5)	(2.22)					8190	5.25	7947	5.47
								(4)	(4.42)
Asphalt F		Asphalt G		Asphalt H		Asphalt I		Asphalt J	
3.5	3.98	3.5	5.40	3.8	5.13	4	7.54[a]	4	12.3
24	4.29	24	5.45	24	5.51	25	7.80	26	15.1
48	4.48	52	5.61	72	5.71	52	8.21	52	16.3
75	4.62	144	5.71	171	5.94	77	8.56	100	18.8
148	4.80	312	5.75	507	6.52	148	8.89	171	21.7
240	5.04	916	5.67	1012	6.71	317	9.38	700	31.4
507	5.30	1053	5.71	2043	6.97			1850	41.9
		2523	5.97	5042	7.42			(4)	(11.4)
		5018	6.19	7251	7.89				
		6528	6.38	(4)	(6.12)				

[a] This value was not used in calculating the line shown in Fig. 1.

with the reversible, less stable changes in structure which are the cause of the age-hardening phenomenon.

The data obtained for the ten asphalts are recorded in Table II and are shown graphically in Fig. 1 where log-viscosity is plotted against the time on a logarithmic scale. No data beyond 1000 hr. are shown on Fig. 1 because to include the longer times would have required the use of a small scale, thereby reducing the clarity of the figure. The lines shown were calculated by the method of averages.

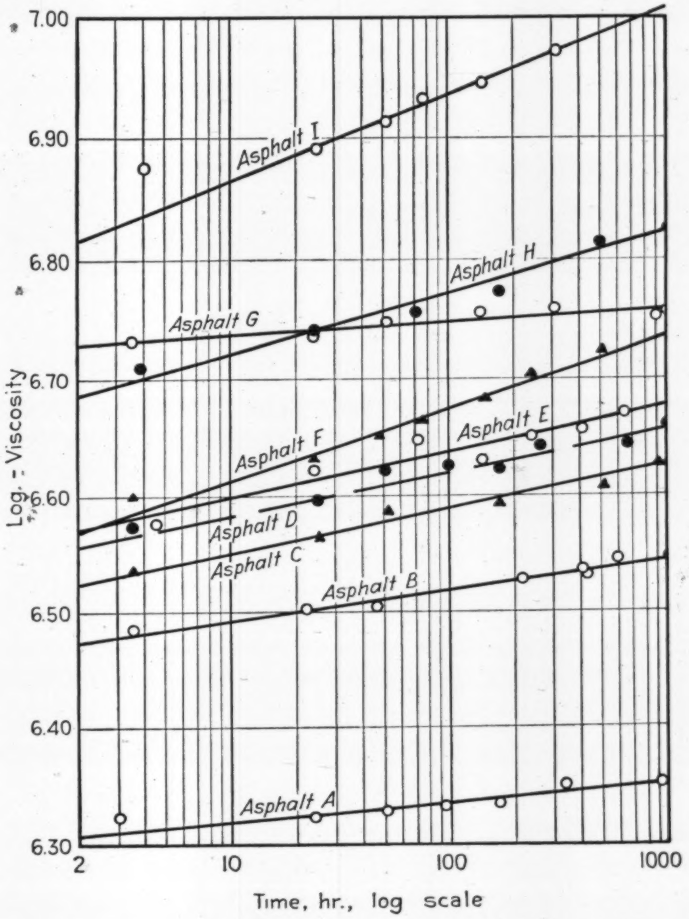

FIG. 1.—Viscosity Data for the Hardening of Asphalt with Time at 25 C. (77 F.).

Data for asphalt J, which is an air-blown bitumen are given in Table II but are not plotted on Fig. 1. This asphalt exhibited an abnormally fast rate of time-hardening which necessitated the use in the later experiments of a shearing stress (12,000 dynes per sq. cm.) which was higher than that used with the other asphalts; the inner cylinder was moved only 0.29 cm. The data for this air-blown bitumen do not give a straight line on either an arithmetic or a log-log plot but do give a smooth curve.

FILLED BITUMENS

An ordinary limestone filler was added to an equal weight of bitumen in order to determine whether the addition of a mineral powder to an asphalt affected the rate of age hardening. Asphalts A, D and I of Table I were the bitumens compounded with the limestone dust.

In this study seven to ten small falling coaxial cylinder viscometers each with a length of 2.540 cm., radius of inner cylinder 1.270 cm., and inside radius of outer cylinder 1.905 cm. were filled with the bituminous mortar and the same general procedure followed as described above for the asphalts. These mortars were all viscous but because their viscosities were considerably higher than for the corresponding asphalts, it was desirable to use a higher mean shearing stress (21,400 dynes per sq. cm.). The distance of movement of the inner cylinder was 0.29 cm.

The limited data obtained from these three series of experiments indicates that age-hardening of a filled asphalt is the same as that of the bitumen present, since the log-viscosity *versus* log-time curves of the two are essentially parallel.

AGING INDEX

The slopes of the various lines shown in Fig. 1 give quantitative measures of the rates of hardening. In an effort to compare this property as possessed by various asphalts an index has been developed based upon the percentage rate of change of viscosity with time.

The straight lines in Fig. 1 are represented by the equation:

$$\eta = bt^m \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where η = viscosity in poises at 25 C. (77 F.),
t = time of aging in hours,
m = slope of the log-log plot, and
b = intercept on the log-viscosity axis.

The rate of change of η with respect to t is not constant but is a function of the viscosity and the time at which the rate is taken, since

$$\frac{d\eta}{dt} = mbt^{m-1} = \frac{mbt^m}{t} = \frac{m\eta}{t} \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

However, by dividing both sides of Eq. 2 by η and multiplying by 100 we obtain the percentage rate of change of viscosity at any time t:

$$\frac{d\eta}{dt} \cdot \frac{100}{\eta} = 100\,\frac{m}{t} \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

It is proposed that 100 hr. be established arbitrarily as the time t at which the asphalts be compared. Within that time sufficient determinations of the viscosity can be made at the proper intervals to calculate accurately the rate of change at 100 hr. Assuming 100 hr. as the basis for comparing various asphalts, a simplified expression results from Eq. 3 which may be called the Asphalt Aging Index, or

$$\text{A.A.I.} = m \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

where m is the slope of the log-viscosity *versus* log-time curve. The proposed index indicates quantitatively the variation in the age-hardening of different types of asphalt. The values of the A.A.I. for the various asphalts are given in Table III.

Discussion

The results presented above show definitely that there is an increase in the viscosity of an asphalt when it is allowed to remain undisturbed at constant temperature and that the age-hardening is largely destroyed by heating. The rate of increase is very rapid immediately after the material has cooled, but slows up as the time increases. It is possible that even on a log-log plot the line is curved in the region up to 3 hr. after pouring. However, it is impossible to measure accurately the viscosity at less than 1 or 2 hr. because the exact time at which the age-hardening phenomenon starts is indeterminate and the sample must be brought to equilibrium conditions with respect to temperature before a determination can be made. This probably explains why in several cases the first determinations are not in line. No evidence has been found to date which indicates that the log-viscosity *versus* log-time relationships for steam- and vacuum-refined asphalts are other than straight lines up to 3000 hr. Thus, the age-hardening characteristics are definitely determined by observations up to 1000 hr.

Table III.—Asphalt Aging Indices for Various Asphalts.

Designation	Source	A.A.I.
G	Californian	0.012
A	Californian	0.018
B	Trinidad	0.026
D	Venezuelan	0.037
C	Fluxed Refined Trinidad Lake Asphalt	0.038
E	Mid-Continent	0.039
H	Mexican	0.051
F	Venezuelan	0.063
I	Venezuelan	0.071

The progressive increase in the viscosity of asphalts discussed above is probably caused by the formation of a structure within the bitumen which is not completely destroyed by the mechanical working incident to the determination of the viscosity in a falling coaxial cylinder instrument. However, a portion of the structure is rather easily broken down since the initial application of shearing stress (first "out" determination) always gave an abnormally high viscosity value, although subsequent values obtained under the same shearing stress, for a particular sample, gave equilibrium values that agreed within the limits of experimental error. Continued shearing of a time-hardened asphalt in a rotating cylinder viscometer indicated that the structure developed in the material may be partially destroyed by continued mechanical working. However, it is impossible to put into the asphalt as much energy by mechanical means as is done by the application of heat and therefore only a small amount of the increased viscosity caused by aging the sample can be eliminated by working the bitumen in a viscometer.

The question will be raised immediately whether or not this age-hardening phenomenon is caused by loss of the more volatile constituents of the asphalt. The reheating *in situ* of a sample which has time-hardened for several months with the return to within a few per cent of the initial low viscosity shows definitely that evaporation of volatile hydrocarbons is not the primary cause of the increased viscosity. When a sample has time-hardened for only a few hundred hours it may be returned by careful heating to its original viscosity. However, with most asphalts the loss of volatile constituents may be an additional factor in the hardening as is evidenced by some of the data obtained after an extended period of time. A permanent change such as this probably explains why reheating after aging for a long period of time does not return the asphalt to its original consistency.

Acknowledgment.—The authors are indebted to Mr. F. B. Burns for valuable contributions during the early part of the investigation. Mr. C. E. Coombs has also made many measurements and has given helpful suggestions.

DISCUSSION

Mr. Prévost Hubbard.[1]—This paper is significant in connection with true heterogeneity. I raise the question as to whether time hardening when reversible upon reheating may not directly measure the heterogeneity of a material as it exists. In this connection I am wondering whether any data have been collected to correlate the heterogeneity test developed by Mr. Oliensis,[2] with time hardening. Has it been found that those materials which react positively to Mr. Oliensis' test show more evidence of time hardening than those which react negatively?

Mr. T. H. Rogers.[3]—I am interested in the nature of the change that takes place. Apparently it is not a chemical change and I am wondering whether there is evidence of departure from viscous flow as a result of aging. Did the authors investigate the viscosity at various rates of shear?

Mr. R. N. Traxler.[4]—We have not made the correlation Mr. Hubbard suggests. However, we know, in general, that highly asphaltic and homogeneous materials show a low rate of time hardening. An air-blown asphalt (for example, asphalt J) time hardens faster and to a greater extent than steam or vacuum-refined asphalts. Thus, the homogeneity or heterogeneity of an asphalt may be indicated by its time-hardening characteristics.

As time hardening develops in an apparently viscous asphalt there must be some departure from truly viscous flow because the phenomenon is apparently dependent upon the building up of a thixotropic structure within the material. Asphalt frequently exhibits the anomalous flow properties commonly encountered in colloidal material.[5] Structural viscosity is not strongly manifested by some of these asphalts; they appear to be viscous liquids although they do deviate to the extent of showing the ability to time harden. Certain asphalts possess structure which can be removed by mechanical working resulting in a viscous material. The air-blown asphalts are definitely non-Newtonian liquids at atmospheric temperatures and the viscosity values obtained are dependent on the shearing stress and rate of shear employed.

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[1] Chemical Engineer, The Asphalt Inst., New York City.

[2] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—a Quantitative Method," see p. 494.

[3] Assistant Director of Research, Standard Oil Co. (Indiana), Whiting, Ind.

[4] Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.

[5] R. N. Traxler and C. E. Coombs, "The Colloidal Nature of Asphalt as Shown by Its Flow Properties." *Journal of Physical Chemistry,* Vol. 40, December, 1936.

COMPRESSION TESTING OF ASPHALT PAVING MIXTURES

BY ROLAND VOKAC[1]

SYNOPSIS

In a brief digest of contemporary tests developed for the study of asphalt paving mixtures it is apparent that heretofore it was not considered feasible to test asphalt paving mixtures by the ordinary methods of compression testing. This paper presents information to show the utility of such ordinary compression testing methods in this field. On the basis of data presented such fundamental characteristics as modulus of elasticity in compression, compressive strength, and elastic limit may be readily evaluated. It is indicated that these physical characteristics may be interpreted in their usual sense by the engineer for the purpose of specification and design.

Two methods of testing have been studied: (1) applying loads for a uniform rate of deformation, and (2) applying a uniform rate of increase of load on a specimen. The former method is found the most practical, and 0.05 in. per min. has been used most successfully. In the ordinary testing procedure decided upon in this work a study of the effects of temperature indicate that room temperatures, about 78 F., are suitable and slight variations do not greatly affect the data.

The effect of diameter and height of test specimens has been studied and as a result it has been found necessary to limit the height to not less than three-fourths the diameter. This is because of an "end effect" on the modulus of elasticity when shorter specimens are used. The effect of compression as measured in the density of a given mixture is also considered. With such treatment of the data it is indicated, for instance, that the stress characteristics of samples with miscellaneous heights and densities as obtained in coring a pavement surface, may be evaluated on a strictly comparable basis.

Equipment necessary for suitable use of the testing methods described may be any compression or universal type testing machine. It is necessary only that the machine will operate to give a fairly constant rate of deformation and can be so operated as to produce stress-strain diagrams by any of the ordinary means.

During the past fifteen years a number of investigators have undertaken the development of tests which offer some measure of control over the physical characteristics of asphalt paving mixtures. This work has resulted in several more or less empirical tests which are peculiarly applicable to testing asphalt paving mixtures. In a paper published in 1925,[2] Hubbard and Field enumerated an impact test by Besson, rod penetration by McNaughton, ball penetration by Howe, compression under static load by Ulman and Milburn individually, a squeeze test by Emmons and Anderton, the shear test by Skidmore, and the stability test for asphalt mixtures by Hubbard and Field. To this list we might add the modifications of the Hubbard-Field stability test by West,[3] the more recent studies of Stokes and Zapata[4] to evaluate the cohesion in asphaltic mixtures sheared under varying normal loads, the work of the California Department of Highways in determining lateral pressures in asphalt paving mixtures under vertical loads, the bearing test in course of development by Berry and Housel,[5] a flexure test at low temperature by Rader,[6] the "model" size test track of Kriege and Gilbert,[7] the larger test tracks of the National Crushed Stone Association,[8] U. S. Bureau of Public Roads and Pennsylvania State Highway Department, and a test measuring lateral flow of a mixture beneath a rotating wheel assembly by Tarwater.[9]

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[1] Chief Engineer of Paving Research, Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.

[2] Prévost Hubbard and F. C. Field, "Researches on Asphalt Paving Mixtures," The Asphalt Assn. *Circular No. 34,* October, 1925.

The Skidmore shear test was the first to measure a single physical property commonly used by the engineer in the evaluation of materials of construction. More recently Rader has developed a method for the evaluation of modulus of elasticity in flexure and modulus of rupture at low temperatures.

In glancing over this array of tests it is at once evident that in general the idea has been to treat asphalt paving mixtures as a material apart or radically different from the ordinary materials of construction. This idea is perhaps justified to some extent. However, in view of our modern concepts of mechanics of materials it is considered that all of our so-called rigid masonry, steel work and the like are subject to some flow under continued application of stress. It is only a matter of degree whether the flow can be perceived in a century, a decade, or a day. The development of modern alloys for instance is making it necessary to revise completely our concepts of elasticity as based upon Hooke's law. We do not always find the constant relation of stress to strain required for this type of elasticity.[10] It may, therefore, be assumed that asphalt paving mixtures do not differ except in degree from other materials of construction. Physical testing of asphalt systems at atmospheric temperatures is analogous to testing metals near their melting points.

The application of a compression test to asphalt paving mixtures has no doubt been delayed by the fact that the values obtained on asphalt compositions are so utterly different in magnitude and character from those usually encountered in materials of construction such as steel and concrete. In this paper we offer data to show that the conventional compression test may be used for the study of asphalt mixtures. We have made an effort to evaluate the effect of specimen dimensions, rate of loading and deformation, and temperature in order that these factors may either be eliminated or standardized in making compression tests on asphalt mixtures.

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[3] W. C. West, "A Ten-ton Roller in the Laboratory and How it Works," *Proceedings*, Assn. Asphalt Paving Technologists, December, 1928, p. 26.

[4] C. R. Stokes and Joseph Zapata, "Devising a Test for Stability of Bituminous Cold Mixtures," *Proceedings*, Assn. Asphalt Paving Technologists, January, 1932, p. 105.

[5] D. S. Berry, "Stability of Granular Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 491 (1935).

W. S. Housel, "A Penetration Method of Measuring Soil Resistance," *Proceedings*, Am. Soc. Testing Mats. Vol. 35, Part II, p. 472 (1935).

[6] L. F. Rader, "Investigations of the Physical Properties of Asphalt Mixtures at Low Temperatures," *Proceedings*, Assn. Asphalt Paving Technologists, January, 1935, p. 49.

[7] H. F. Kriege and L. C. Gilbert, "Some Factors Affecting theResistance of Bituminous Mixtures to Deformation Under Moving Wheel Loads," *Proceedings*, Assn. Asphalt Paving Technologists, December, 1933, p. 73.

[8] A. T. Goldbeck, "Tests for the Traffic Durability of Bituminous Pavements," *Proceedings*, Assn. Asphalt Paving Technologists, January, 1936, p. 44.

[9] E. L. Tarwater, "A Roller Testing Machine for Measuring the Stability of Bituminous Mixtures," Division of Tests, Bureau of Public Roads Report.

[10] L. B. Tuckerman, "Aircraft: Materials and Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 31 (1935).

DESCRIPTION OF EQUIPMENT

For running compression tests on asphaltic mixtures we have used a 60,000-lb. Baldwin-Southwark hydraulic compression machine. The lower head on the testing machine is part of the hydraulic ram and is opposed by an adjustable cross-head with a spherically seated bearing plate. Three load-indicating dials cover ranges from 0 to 5000, 0 to 30,000, and 0 to 60,000 lb., respectively, and any one or all three may be used according to the range of material strengths involved. Each dial is equipped with a load-pacing disk by means of which a constant rate of loading may be applied to the test specimen by manual control of the feed from the pump into the ram; the feed is adjusted so that the pointer on the indicating dial moves uniformly with any set speed of the pacing disk. The speed of the pacing disk may be adjusted to correspond to rates of loading from 300 to 60,000 lb. per min.

In the course of our earlier experimental work we often used a fixed setting on the feed-control valve as approximating a constant rate of movement of the ram and therefore deformation of the specimen. We have since constructed a simple mechanism which translates the vertical movement of the ram into a circulating motion of a pointer concentric with the load-pacing disk on the 60,000-lb. dial. By means of this device we may accurately control the rate of deformation at any speed from 0.0075 to 0.3000 in. per min.

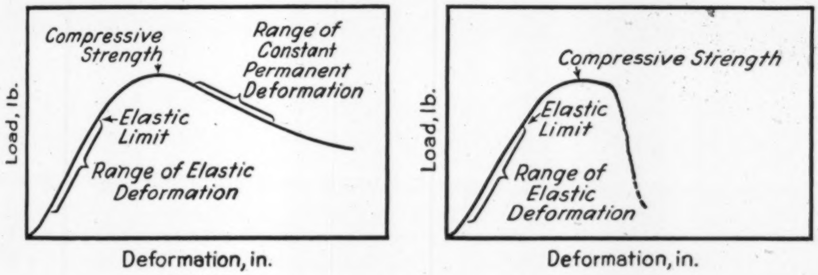
Load-deformation curves are obtained with a Southwark-Templin automatic stress-strain recorder and a Southwark-Peters extensometer. This equipment furnishes a permanent autographic record of load-deformation characteristics of every sample tested.

We have investigated the phenomena to be discussed with specimens made in four sizes of molds; however, in order to demonstrate the effect of the dimensions of test specimens, data from only two different sizes of molds will be used. These molds are (1) the usual 2-in. diameter Hubbard-Field mold, and (2) a mold for briquets 1 sq. in. in cross-section (1.13 in. in diameter). Each mold is equipped with two opposed plungers between which the briquet is compressed while the mold itself is in a free-floating position.

The molds are sufficiently tall that briquets may be made with heights twice their diameter when required. In order to make briquets of definite height, gages were made of $\frac{3}{16}$-in. drill rod to establish the required distance between the bearing plate and ram of the press, allowing space for the specimen and upper and lower opposed plungers. These rods were made in proper lengths for specimens whose ratio of diameter to height would be 4.00, 2.00, 1.33, 1.00 and 0.66, respectively.

METHODS OF TESTING

A specimen to be tested is centered on the ram and the cross-head is lowered until the hemispherically seated bearing plate it carries just touches the specimen. The specimen is then "seated" under a load of about 100 lb. per sq. in. at the rate of loading or deformation to be used in the test, after which the load is immediately released. This serves to smooth out any irregularities of the two parallel faces of the specimen and results in a better stress-strain curve in the lower range of deformation.



(a) Constant rate of deformation. (b) Constant rate of loading.

FIG. 1.—Typical Load-Deformation Curves.

The extensometer is then adjusted, the recorder is put into operation, and the test is run to completion. As stated previously, there are two methods of testing that may be used: (1) the specimen may be subjected to a constant rate of deformation, or (2) a constant rate of loading.

At a uniform rate of deformation the load-deformation curve has the appearance of Fig. 1 (*a*). At a constant rate of loading the load-deformation curve has the form shown in Fig. 1 (*b*). In Fig. 1 (*a*) the curve shows a straight-line relation of load to deformation initially. Then there is a deviation from the straight line showing a decrease in increments of load as deformation continues until a maximum load is reached. The load then starts to decrease at an increasing rate as deformation continues entering another region where load is proportional to deformation, after which the decrease in load diminishes causing the curve to flatten out. In Fig. 1 (*b*) the same changes occur until the maximum load is reached when deformation becomes so rapid as to make it impossible to maintain constant rate of loading.

Manual control of constant rate of loading in the region of maximum load is extremely difficult and the autographic record of load-deformation may become quite erratic. No such difficulty is encountered in testing at a constant rate of deformation. In our ordinary testing procedure, therefore, we run all tests at a constant, or in some cases approximately constant, rate of deformation. A fixed setting of the manual-control valve gives a fair approximation of constant movement of the ram even under load since our testing machine is equipped with an automatic compensating device for this purpose. For the purpose of research we use the finer control obtained with the deformation-pacing disk.

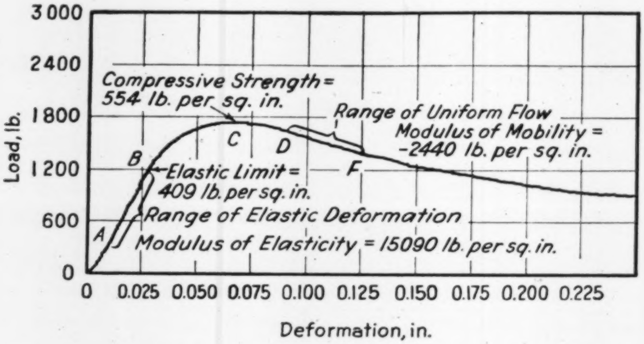


FIG. 2.—Typical Autographic Load-Deformation Chart.

Briquet, 2 in. in diameter by 1 in. in height. Temperature of test, 78 F. Rate of loading, 0.050 in. per min.

CHARACTERISTICS IN A TYPICAL STRESS-STRAIN CHART

In Fig. 2 is shown a typical autographic chart as produced by the Southwark-Templin stress-strain recorder, which is a load-deformation curve in that load is plotted against deformation rather than stress against strain. It is possible by adjustment to make the ordinate units represent 200, 400, 600 or 800-lb. load within the 5000-lb. range ordinarily used. The abscissa units may represent 0.025, 0.050, or 0.100 in. deflection according to the adjustment made in the recorder. The specimen tested was 2 in. in diameter by 1 in. high; the temperature was 78 F., and the rate of deformation was 0.05 in. per min.

There is a slight curvature at the beginning of the graph while the specimen is assuming the load. Then from points *A* to *B* the curve is a straight line, showing a proportional relationship of load to deformation (or of stress to strain) or, in other words, a range of elastic deformation. From point *B* the curve deviates from the straight line and shows a decreasing increment of load for equal increments of deformation up to the maximum load at point *C*. The stress calculated at point *B* is referred to in this paper as the elastic limit and that at point C as the compressive strength, or ultimate stress. Beyond point C the curve shows negative increments of load with continued deformation to point D where the load-deformation relationship again becomes a constant value to point F. This constant represents in some manner the flow or mobility characteristics of the mixture. We tentatively describe the negative ratio of stress to strain represented by this straight line as the "modulus of permanent deformation." In a discussion of Rader's paper[11] before this Society last year we mentioned this characteristic evaluated in our test as the "modulus of mobility." The present name is preferable in order to avoid conflict with the strict definition of "mobility" used by the rheologists. Beyond point F the negative increments of load become smaller with continued deformation and the curve shows a tendency to flatten out.

A comparison of the condition of the specimen under test with these several portions of the curve is enlightening. Up to point B the effect of loading is to cause deformation only in that amount necessary to develop the full measure of resisting stress due to the elastic deformation of the cementing medium. However, as soon as the curve starts to deviate from the straight line at the elastic limit, bulging of the sample becomes more noticeable, and increases as deformation is continued. This would seem to indicate that the shear strength of the cementing medium has been exceeded and permanent deformation will result. The specimen has become decidedly barrel-shaped after reaching the compressive strength at point C, though there is no change in the area of contact with the ram and bearing plate of the testing machine. The barrel shape becomes more pronounced throughout the range from points D to F, and at point F the contact areas with the ram and bearing plate become greater, continuing to increase thereafter as deformation continues.

It is our experience with specimens of many different mixtures that the straight line portion A–B represents elasticity of the asphalt mixture tested. Tests have shown that elastic recovery does take place when the load at point B (the elastic limit) is not exceeded. It is true that there is considerable lag in this recovery resulting in a very broad hysteresis loop but within a day the deformation will have disappeared and the only permanent change in height of the specimen will be the amount represented by the difference between the intersection of line A–B prolonged to the X-axis and the original origin. This loss in height is measurable with a micrometer and represents only the slight rearrangement of the surface grains of aggregate in the mix as they orient themselves for better contact with the bearing surfaces.

Using the values of load, deformation and size of specimen recorded in

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[11] Discussion by Roland Vokac of paper by L. F. Rader, 'Investigations of the Physical Properties of Asphaltic Mixtures at Low Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 572 (1935).

Fig. 2, the several characteristics of the tested mixture may be evaluated as follows:

Modulus of Elasticity.—The modulus of elasticity is by definition the stress divided by the strain within the elastic range. Therefore, the slope of the straight line *A–B* in terms of the two axes and the dimensions of the test specimen is the modulus of elasticity and may be evaluated by the usual procedure for calculating the modulus in terms of the slope of the curve *A–B* between two points in the elastic range, expressed as unit stress and unit strain by introducing the dimensions of the test specimens. In the present instance, the value reduces to 15,090 lb. per sq. in.

Elastic Limit.—The elastic limit is the unit stress at point *B* where the curve *ABCDF* starts to deviate from the straight line *A–B*, and is the load recorded at this point divided by the original cross-sectional area. For example, the elastic limit in the present instance is $\frac{1284}{3.14}$ or 409 lb. per sq. in.

Compressive Strength or Ultimate Stress.—The compressive strength of the specimen is the ultimate stress recorded, namely at point *C*, calculated by dividing the load at this point by the cross-sectional area. It reduces to 554 lb. per sq. in.

"Modulus of Permanent Deformation."—The "modulus of permanent deformaion" is measured by the negative slope of the straight-line portion *D–F* of the curve and is evaluated in a manner similar to the modulus of elasticity. It is the change in stress divided by the change in strain and reduces to -2440 lb. per sq. in.

The "modulus of permanent deformation" is an interesting factor related to the type of flow that occurs with continued deformation after the compressive strength has been reached. We have found it to be susceptible to the rate of loading or rate of deformation, the dimensions of the specimen tested, and the temperature of test. It becomes larger as the rate of loading or of deformation is increased, and smaller with an increase in temperature.

Elastic Limit Establishes Safe Working Stress:

The elastic limit appears to have a more or less constant relationship to the compressive strength in the materials studied. Its value is generally 0.60 to 0.75 of the compressive strength in all mixtures considered, and is therefore in a general way amenable to the same treatment that is to be given the compressive strength. It is to be recognized, however, that this constancy of relationship to the compressive strength is important from the standpoint of design. It fixes the value of a factor to be applied to the compressive strength if the elastic limit is not to be exceeded. In other words, if a mixture has a compressive strength of 420 lb. per sq. in., the safe working stress will be 0.60 of this, or 252 lb. per sq. in. Or conversely, if a surface is to bear loads of say 240 lb. per sq. in., a mixture should be furnished with compressive strengths of not less than 400 lb. per sq. in.

TABLE I.—OBSERVED DATA ON 2 AND 1.13-IN. DIAMETER SPECIMENS.

	Values of p for various D/h Ratios and Densitv			
	Density, 1.748	Density, 1.941	Density, 2.138	Density, 2.33
(a) p MEASURED IN SPECIMENS 2 IN. IN DIAMETER (3.14 SQ. IN. AREA)[a]				
$h = 2.00$ $D/h = 1.00$	69	148	338	517
$h = 1.50$ $D/h = 1.33$	87	186	420	714
$h = 1.00$ $D/h = 2.00$	168	323	636	1088
$h = 0.75$ $D/h = 2.66$	239	439	819	1450
(b) p MEASURED IN SPECIMENS 1.13 IN. IN DIAMETER (1 SQ. IN. AREA)[a]				
$h = 1.710$ $D/h = 0.66$	51	126	248	444
$h = 1.113$ $D/h = 0.99$	71	151	364	534
$h = 0.844$ $D/h = 1.33$	90	192	408	720
$h = 0.564$ $D/h = 2.00$	174	283	618	1141
$h = 0.424$ $D/h = 2.66$	258	442	820	1515

[a] Ratios of values of p for 2-in. specimens to values for 1.13-in. specimens are constant and approximately equal to 1.00, as follows:

	Ratios of $p_{2\text{ in.}}/p_{1.13\text{ in.}}$			
	Density, 1.748	Density, 1.941	Density, 2.138	Density, 2.33
$D/h = 1.00$	0.97	0.98	0.93	0.97
$D/h = 1.33$	0.93	0.97	1.03	0.99
$D/h = 2.00$	0.97	1.14	1.03	0.95
$D/h = 2.66$	0.93	0.99	1.00	0.95

TABLE II.—OBSERVED DATA ON 2 AND 1.13-IN. DIAMETER SPECIMENS.

Values of modulus of elasticity.

	Values of E for various D/h ratios and density shown[a]			
	Density, 1.748	Density, 1.941	Density, 2.138	Density, 2.33
(a) E MEASURED IN 2-IN. DIAMETER SPECIMENS				
$h = 2.00$ $D/h = 1.00$	3290	6130	9 000	15 000
$h = 1.50$ $D/h = 1.33$	3260	5950	9 250	15 510
$h = 1.00$ $D/h = 2.00$	3150	6150	10 150	16 790
$h = 0.75$ $D/h = 2.66$	3680	6000	17 160	22 000
$h = 0.50$ $D/h = 4.00$	3660	8560	32 050	30 600
(b) E MEASURED IN 1.13-IN. DIAMETER SPECIMENS				
$h = 1.113$ $D/h = 1.00$	3270	6360	10 200	16 410
$h = 0.844$ $D/h = 1.33$	3110	5980	9 600	16 210
$h = 0.564$ $D/h = 2.00$	3460	6320	10 100	16 690
$h = 0.424$ $D/h = 2.66$	3340	6260	18 650	22 100
$h = 0.282$ $D/h = 4.00$	5040	9210	......	32 700

Average of E for D/h = 1.33 or less:

	Density, 1.748	Density, 1.941	Density, 2.138	Density, 2.33
Average E when D/h is not greater than 1.33	3230	6105	9 515	15 780

Effect of Dimensions on Physical Characteristics:

In order to limit the variables in the test specimens to those of diameter and height alone, briquets were made of the same sheet asphalt mixture. However, since even in the same mixture, the structure due to differences in compaction or density may vary and thus cause variations in the resisting stresses, it was necessary carefully to compress each set of briquets used to definite sizes resulting in equal densities. Four such sets were made as listed in Tables I and II with densities of 1.748, 1.941, 2.138 and 2.33, respectively, in order to note the effect of this variable. At each of these densities briquets 2.00 and 1.13 in. in diameter were made with heights necessary to give a ratio of diameter to height $\frac{D}{h}$ equal to 0.66, 1.00, 1.33, 2.00, and 2.66. Thus, in Tables I and II there are really four sets of data each comparable except for density.

Effect of Dimensions on Compressive Strength:

Considering each set of data separately and plotting the compressive strength, p, against the ratio of diameter to height, $\frac{D}{h}$, for both the 2- and 1.13-in. diameter briquets, produces the four radiating straight lines in Fig. 3. Note the satisfactory agreement of data for the two diameters of briquets tested. Each of these lines, therefore, represents the relationship of p to $\frac{D}{h}$ for this mixture in a given state of compaction. For other mixtures the slopes of these lines would probably be different. It may be said accordingly that the four radiating lines show the relationship of p to $\frac{D}{h}$ for as many different mixtures.

The general equation of these lines is

$$p = m\left(\frac{D}{h}\right) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

where p = compressive strength of this mixture (at a given density) in pounds per square inch,

$\frac{D}{h}$ = ratio of diameter to height, and

m = a constant for this mixture (at a given density) and is the slope of a line relating p to the ratio of $\frac{D}{h}$.

The values of m for each density are shown in the equations in Fig. 3.

The general Eq. 1 indicates that in any mixture the compressive strength will be dependent upon the ratio of diameter to height. Thus,

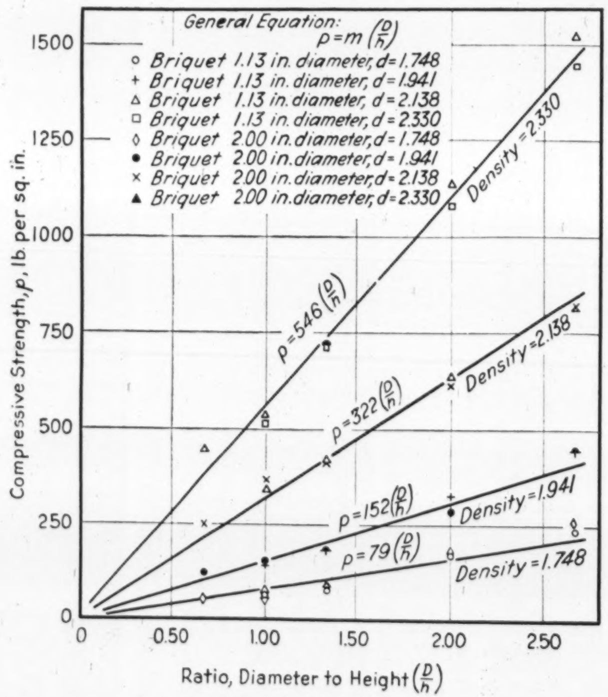

FIG. 3.—Relationship of Compressive Strength to $\frac{D}{h}$.

Illustrated with four sets of data representing four densities of the same mixture.

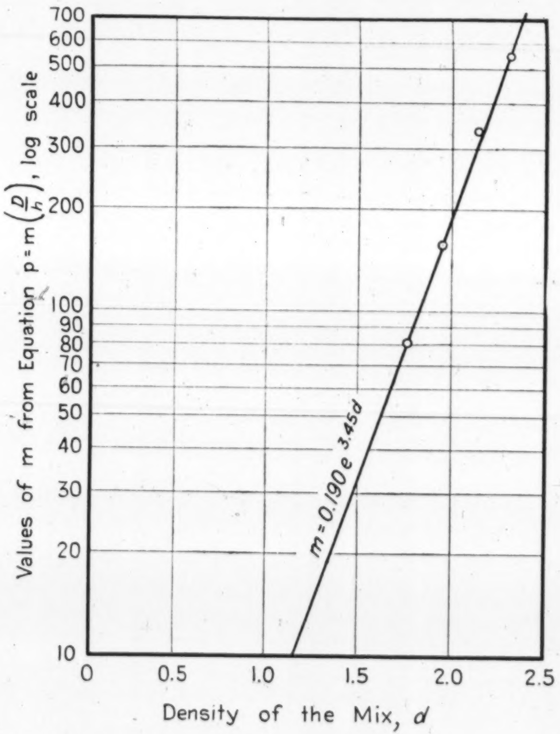

FIG. 4.—Relationship of m to the density.

General equation $m = a\,e^{bd}$

doubling the height of a specimen, all other things remaining equal, will halve the compressive strength. Eq. 1 is most practical for direct comparison of strengths of mixtures since it will be noted that the compressive strength of mixtures is directly proportional to m for equal $\frac{D}{h}$ values.

Effect of Density on Compressive Strength:

Since the mixture used is identical in each of the four sets of data the difference in slope of the four lines is entirely due to structural arrangement of the mixture composition resulting from the four densities listed in Table I. The values of slope m can therefore be related to the density of this mix. The four values of m from equations evaluated in Fig. 3 and the corresponding densities have been plotted in Fig. 4 showing an exponential relationship of the form:

$$\log m = \log a + (b \log e)\, d$$

or,

$$m = a\, e^{bd} \quad \text{.......................} (2)$$

where d = the density of the mix,
a and b = numerical constants, depending upon the structure and composition of the mix, and
e = base of Naperian logarithms.

Evaluating Eq. 2 with the data shows

$$\log m = 1.498\, d - 0.723$$

and,

$$m = 0.190\, e^{3.45\, d}$$

The right-hand side of Eq. 2 may be substituted in Eq. 1 to obtain the most general equation

$$p = a\, e^{bd} \left(\frac{D}{h}\right) \quad \text{.....................} (3)$$

This is perhaps the best form in which to use the relationship of $\frac{D}{h}$ and p for practical purposes. In this study all variables except the dimensions of the specimens had to be held constant, so it was necessary to compress cylinders to exact, known densities. However, with Eq. 3 available, data from any two or more dissimilar briquets of the same mix tested after measurement of size and density are enough to evaluate the two constants

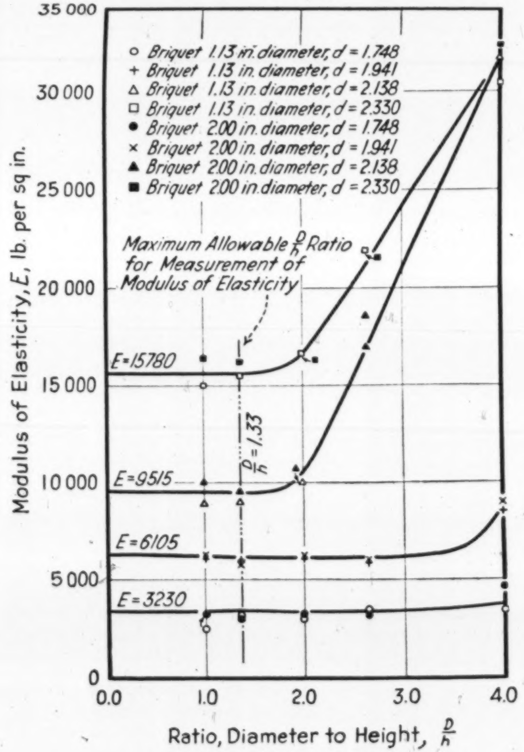

FIG. 5.—Relationship of E to $\frac{D}{h}$.

Four sets of data representing four densities of the same mixture. Shows constant values of E at $\frac{D}{h}$ less than 1.33.

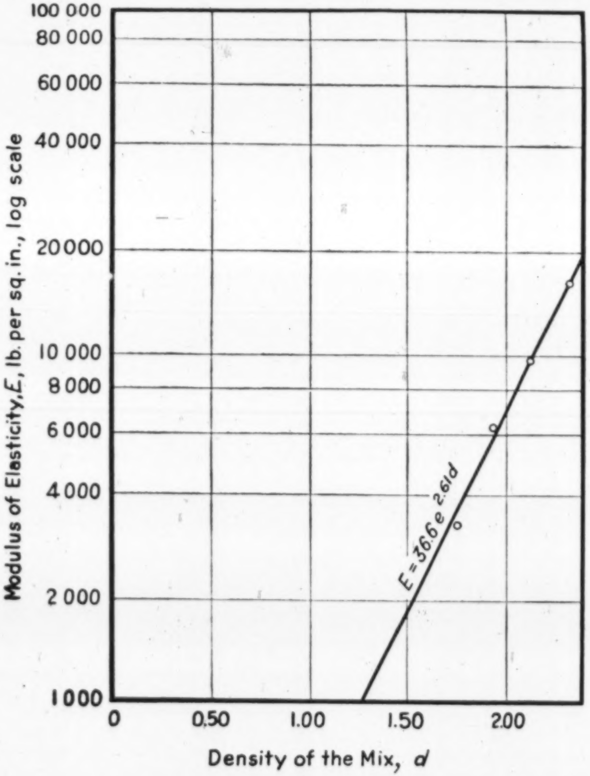

FIG. 6.—Relationship of E to the Density of the Mix.

General equation: $E = a'e^{b'd}$

a and b in Eq. 3 and to obtain the constant m in Eq. 1. This materially simplifies the technique required in the laboratory for routine design procedure.

Effect of Dimensions on the Modulus of Elasticity:

Referring to the data in parts (*a*) and (*b*) of Table II, it is at once evident that the modulus of elasticity is constant for each of the four densities of mix when the $\frac{D}{h}$ ratios are small.

According to Hooke's law the ratio of stress to strain is a constant in the elastic range and is not affected by dimensions. Our test data in Table II, however, indicate that we may expect deviations from this constant value if certain ratios of dimension are exceeded. It appears, therefore, that the modulus of elasticity is influenced by some kind of "end effect" during the process of testing which is detected when the specimen used is disk-like rather than cylindrical.

In Fig. 5 all of the data from Table II have been plotted showing the relationship of E to $\frac{D}{h}$ for all four densities and the two diameters used. The flat portion of the curves in the region of lower values of the $\frac{D}{h}$ ratio is at once evident. The influence of shape is seen in the higher moduli of elasticity measured in the short, flat specimens. It is safe to assume from this set of curves that the "end effect" causing these higher moduli will be eliminated from any of the specimens tested if the ratio of diameter to height is held less than 1.33. This means that the height of a test specimen should never be less than three-fourths the diameter of the specimen in order to obtain constant values of the modulus of elasticity for any given density of specimen.

Effect of Density on Modulus of Elasticity:

Using this portion of the data, we find that for each given density the average modulus of elasticity, E, has the value given in footnote *a* of Table II. These values of E have been plotted against density, d, in Fig. 6 to evaluate their relationship. The equation is found to be of the form

$$E = a' e^{b' d} \qquad (4)$$

This equation will satisfy the data for any density of the mix when $\frac{D}{h}$ is not greater than 1.33. The constants a' and b' will differ for any other conditions of test and composition. Evaluating Eq. 4 for the data given results in:

$$E = 36.6\, e^{2.61 d}$$

Effect of Rates of Testing:

Important among the variables that may be introduced in the actual testing procedure itself are the rate of loading and rate of deformation. The effect of rate of loading and rate of deformation on the modulus of elasticity and compressive strength have been determined using the data from Tables III and IV. The equations are:

(*a*) Rate of loading (R_2) (pounds per square inch per minute)

$$p_2 = 141\ R_2^{0.231}$$

$$E_2 = 2120\ R_2^{0.294}$$

(*b*) Rate of deformation (R_1) (inches per inch per minute)

$$p_1 = 1500\ R_1^{0.230}$$

$$E_1 = 56{,}500\ R_1^{0.294}$$

The general forms of these equations are, therefore,

$$p = C\ R^k \qquad (5)$$

and,

$$E = C'\ R^{k'} \qquad (6)$$

Knowing the value of constants C, k, C' and k', it is therefore possible to compare data obtained at any rates of loading or deformation by use of these two equations.

Due to the logarithmic equations that result it should be desirable in testing procedure to use low rates of testing, thereby eliminating the possibility of large errors due to irregularity in control of the speeds of testing.

Effect of Temperature:

The data in Table V have been used to calculate the relationship between the modulus of elasticity and compressive strength, and the temperature of testing. The equations satisfying these data are, for the modulus of elasticity,

$$E = \frac{74{,}650}{e^{0.0156\,T}}$$

and for compressive strength,

$$p = \frac{2320}{e^{0.0182\,T}}$$

where T = temperature in degrees Fahrenheit, and
e = base of Naperian logarithms.

The general forms of these equations are:

$$E = a\,e^{-bT} \quad \text{(7)}$$

and,

$$p = c\,e^{-dT} \quad \text{(8)}$$

where a, b, c and d are constants; e and T are as before.

TABLE III.—EFFECT OF RATE OF LOADING ON COMPRESSIVE STRENGTH AND MODULUS OF ELASTICITY.

	Rate, R_2, lb. per sq. in. per min.							
	95.6	127	159	223	318	382	445	636
Compressive strength, p, lb. per sq. in.	406	448	429	480	517	521	557	534
Modulus of Elasticity, E, lb. per sq. in.	8140	8790	9440	10 400	11 600	12 100	13 200	13 300

TABLE IV.—EFFECT OF RATE OF DEFORMATION ON COMPRESSIVE STRENGTH AND MODULUS OF ELASTICITY.

	Rate, R_1, in. per inch per min.							
	0.010	0.025	0.040	0.055	0.070	0.085	0.115	0.130
Compressive strength, p, lb. per sq. in.	560	648	674	709	828	828	865	941
Modulus of Elasticity, E, lb. per sq. in.	15 700	19 600	19 800	23 300	26 900	27 700	26 600	32 600

TABLE V.—EFFECT OF TEMPERATURE ON COMPRESSIVE STRENGTH AND MODULUS OF ELASTICITY.

TEMPERATURE, T, DEG. FAHR.	MODULUS OF ELASTICITY, E, LB. PER SQ. IN.	COMPRESSIVE STRENGTH, p, LB. PER SQ. IN.
133	7 560	339
123	11 340	425
113	16 640	659
93	17 200	937
80	20 630	878
70	22 800	1243
50	31 780	2002
37	49 810	2479

Knowing the values of these constants, corrections can be made to adjust data to the same temperature basis. Due to the fact that T is part of a negative exponent it is apparent that in the low range of temperatures the effects will be very large, and possibility of error due to small variations in temperature will be greater.

CONCLUSIONS

The data presented show the possibilities in a compression test for asphalt paving mixtures. It has been indicated that the modulus of elasticity and compressive strength are not entirely independent of the dimensions of the test specimen. However, the derived relationships of compressive strength to diameter, height and density of the test specimen are very simple and have a minimum of constants in the general equations. Limiting the shape of the test specimens with a maximum ratio of $\frac{D}{h} = 1.33$ is a rational procedure to insure a proper evaluation of the modulus of elasticity and will not affect the evaluation of constants in the general equations for compressive strength.

From a practical standpoint this limitation of $\frac{D}{h}$ is not very restrictive. It merely requires, for instance, that in the case of a 2-in. diameter specimen the height must not be less than $1\frac{1}{2}$ in.

Correlating the measured physical characteristics with the density of the test specimen is of great practical value. In the ordinary preparation of briquets of dissimilar heights or diameters using the same mixture, it is a difficult procedure to obtain briquets of identical densities. The equations given for E and p in terms of density eliminate the necessity for making all briquets of the same density. This greatly simplifies the laboratory work involved. It furthermore makes it possible to evaluate the constants of a mixture characteristic on the basis of cores cut from a pavement. Such cores are rarely of the same height, or of the same density.

It is not to be supposed that the densities used in the data given in this paper may be considered as an arbitrary basis for other mixtures containing dissimilar aggregates and asphalt cements. The density considered is the density of a mixture resulting from certain states of compaction obtained under different conditions of compression either in specimens or in a pavement sample.

No attempt has been made arbitrarily to specify what rates of loading or deformation should be used in the actual testing procedure. These and the temperature at which the test is to be conducted will more properly be specified after a suitable period of development during which extensive tests should be made under many different conditions with a great variety of mixtures. In the present preliminary study we have had the greatest success testing at room temperature (about 78 F.) at a constant rate of deformation of 0.05 in. per min.

Although the present data were obtained using one of the newest types of hydraulic testing machines, it is entirely possible to use any universal or compression-type testing machine for satisfactory results, provided it will operate to give a fairly constant rate of deformation and can be so operated as to produce stress-strain diagrams by any of the ordinary means.

TESTING FOR SLUDGE FORMATION IN MINERAL TRANSFORMER OIL

By F. M. Clark[1] and E. A. Snyder[2]

Synopsis

With the clear recognition that the successful operation of oil-filled electrical equipment depends to a large extent on the chemical and electrical stability of the oils used, the Society's Committee D-9 on Electrical Insulating Materials has consistently attacked one of the most important phases of the problem—that of sludge formation in oil-filled transformers. The ultimate objective has been the establishment of a sludge-testing procedure capable of exact reproduction and duplicating the chemical effects normally occurring under commercial transformer operation, as a result of which oil-insoluble products, commonly called sludge, are formed. This paper constitutes a summary of the results obtained to the present time. The progress which has been made, upon which the plans for future investigation are to be based, merits the serious consideration of the Society.

The technical investigation described in this paper has been obtained with the co-operative assistance of the following laboratories:

The Atlantic Refining Co., Philadelphia, Pa.
Commonwealth Edison Co., Chicago, Ill.
The DeLaval Separator Co., Poughkeepsie, N. Y.
The Detroit Edison Co., Detroit, Mich.
Electrical Testing Laboratories, New York City.
General Electric Co., Pittsfield, Mass.
Socony-Vacuum Oil Co., Inc., New York City.
Standard Oil Co. (New Jersey), Elizabeth, N. J.
Sun Oil Co., Philadelphia, Pa.
The Texas Company, New York City.
Tide Water Oil Co., Bayonne, N. J.
Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

The Problem and Method of Attack

The goal of the present investigation is the establishment of a testing procedure able to indicate clearly and accurately the sludge-forming characteristics of mineral oil drawn from transformers in actual commercial use. Such a test is essential for the proper inspection of new and unused transformer oil, and for the standardization of such testing procedures as may be adopted for routine control and acceptance work.

The chemical changes resulting from sludge formation in mineral oil are recognized to be affected by the presence of materials generally classed as contaminants. Such materials may include even the products of the reaction itself. In most instances the result is the acceleration of the oxidation and sludge formation, although the possibility of retardation effects cannot be wholly ignored. Because of the variety of chemical changes possible in mineral oil during commercial transformer use, and the tendency of such oil to change even under careful conditions of storage after having been drawn from the transformer, it has been found necessary in the preliminary work upon which the foundation of the detailed test procedure must be built, to use new mineral oil capable of reproduction and storage without change for varying lengths of time. The investigation described in this paper has concerned itself chiefly with the establishment of those testing details which must be followed if reproducible results are to be obtained in the examination of the sludging characteristics of transformer oil. The oils investigated include those of the type which have been established by years of successful use as being satisfactory cooling and dielectric media for transformers. The application of the testing principles developed to the examination of the transformer oil during commercial use has not yet been attempted.

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[1] Chemist, General Electric Co., Pittsfield, Mass.
[2] Socony-Vacuum Oil Co., Inc., New York City.

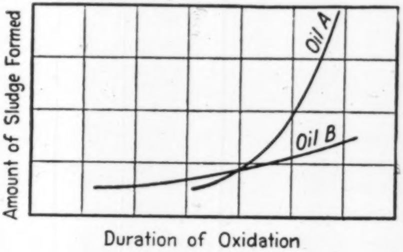

FIG. 1.—Illustrating the Sludge Formation Characteristics Met in the Oxidation of Transformer Oil.

FIRST TRACE OF SLUDGE FORMATION

In order to meet the demands of later application to transformer oils that have been in service, the first requirement laid down for a suitable sludge test was that the results obtained should be capable of positive statement. This effectively eliminated a test based on the total absence of sludge formation in a short but definite time interval of oxidation. The result was the development of what has been frequently referred to as the "life test." In view of its later developed defects, detailed analysis of the background of data accumulated during the development of this test will not be made. The method of carrying out the test has been described in an article by one of the authors, published in the *Proceedings* of the Society.[3] Essentially, this test consists in the oxidation of the oil at 120 C. The oil during oxidation is examined for evidence of sludge formation at daily intervals, the results being expressed in days of oxidation time necessary to produce the first signs of sludge precipitation.

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[3] E. A. Snyder, "Methods for Determining Sludging Properties of Transformer Oils," *Proceedings*, Am. Soc Testing Mat., Vol. 23, Part I, p. 448 (1923).

The "life test" for transformer oils was shown in the early stages of the present investigation to be entirely unsuited for the study of transformer oil in service use. Small amounts of what appeared to be a negligible contaminating material were sufficient to reduce the "life" of the oil from an acceptably high value to one of extremely short duration.

The weakness of the "life test" for transformer oils was the basic assumption that all oils, once oxidation has reached the sludge-formation point, deposit sludge at approximately the same rate. This is not true. When the oxidation of mineral transformer oil is carried well beyond the sludge-formation point, at least two clearly defined types of behavior become apparent. These are illustrated in Fig. 1. Experience with the "life test" has shown that distinction must be made on a basis which will include consideration not only of the resistance of the oil to deposit first traces of sludge, but also the rate at which sludge accumulates after its first appearance.

THE SLUDGE-ACCUMULATION TEST

The sludge-accumulation test is an attempt to eliminate the defects of the earlier "life test." As its name implies, the sludge-accumulation test places special emphasis on the rapidity of sludge deposition after the formation of oil-insoluble products once begins.

In the establishment of the sludge-accumulation type of test, two possibilities are offered. First, a definite time interval of test may be set and the amount of sludge formed in such time interval reported. Such procedure has appeared to be too closely allied with the defects clearly established for the "life test." No information is obtained from such a test to indicate whether the oil at the moment of sampling is rapidly increasing in the rate of sludge precipitation. The second possibility involves the repeated examination of the oil over a period sufficiently extended to indicate clearly the rate change in sludge deposition with prolongation of the oxidation period. It is with this type of testing procedure that the present paper is especially concerned. No attempt is made to establish any maximum rate of sludge accumulation as an accepted value for transformer oil. Effort has been entirely centered on the problem of eliminating those features of the testing procedure which are contributory to the variation of the test results for different oxidation runs in the same or different laboratories.

Oil-Testing Factors:

The factors of test given special attention during the present investigation have included: the testing oven, the testing temperature, the oil receptacle, the rate and conditioning of the oxygen supply, the method of sludge determination, and the use of an oxidation accelerator. These factors of test are discussed in subsequent paragraphs.

The Testing Oven.—Because of the adequate temperature control and the highly desirable mechanical features developed for the oven in the life test already referred to, this type of oven was tentatively accepted as the standard apparatus for the investigation.

In accepting the standard design of the life-test oven, it is recognized that the heating mechanism used will affect the sludge results obtained. The standard oven heats the oil by means of its contact with the hot air passing through the oven in such a manner as to spread out over the top surface of the oil. Figure 2 shows that heated in such a manner, the accumulation of sludge is slower than when heated "from the bottom," thereby promoting oil circulation within the testing vessel. It is further recognized that heating from the top promotes oil evaporation. These testing details must be remembered in the analysis of the sludge-testing data set forth.

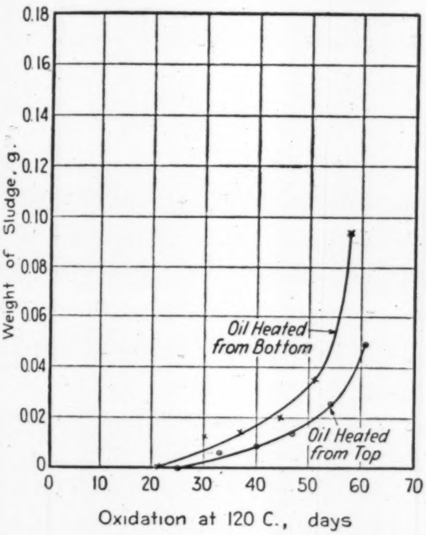

FIG. 2.—Showing the Variation in Sludge Accumulation in Transformer Oil Oxidized at 120 C.

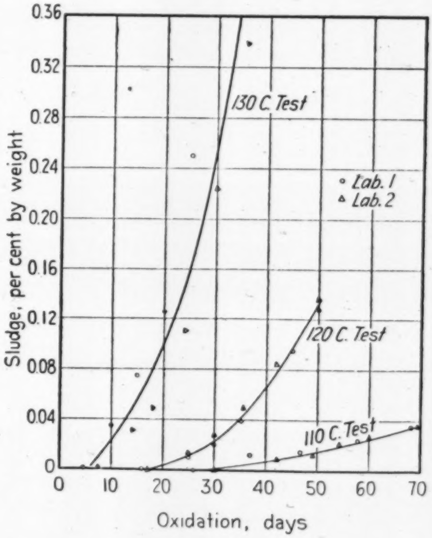

FIG. 3.—Effect of Temperature on Sludge Accumulation in Mineral Transformer Oil.

The Testing Temperature.—It is submitted that the oil oxidation temperature should be as low as practicable in order to eliminate abnormal chemical changes. The minimum allowable temperature was established at 120 C. without copper catalyst, being determined by the length of time necessary to evaluate clearly the sludge-forming properties of the oil. Anticipating the data to be discussed in later paragraphs, it is established that at least 0.05 per cent sludge by weight must be formed in the oxidizing oil in order clearly to define the differences in oil-sludging rate. At 120 C. under the conditions of oxidation outlined, as long as 45 days may be necessary with American transformer oils before a real gage of the sludge-accumulation rate can be secured. With such an extended time factor, it is obvious that lower temperatures than 120 C. are impracticable. Figure 3 compares the sludge accumulation of mineral transformer oil at temperatures between 110 and 130 C. As seen from Figs. 4 and 5, the variation in testing temperature between 110 and 130 C. produces no abnormality which would be indicative of a marked change in the oil-sludging characteristic. Figure 6 illustrates the effect of testing temperature on the first signs of sludge formation. Here again no abnormality in the sludge formation seems evident.

Although no data are obtained indicating that temperatures as high as 130 C. are productive of abnormal results, the temperature of 120 C. was accepted for the oxidation of the oil as being sufficiently high not to prolong the test unduly and yet sufficiently low to evaluate the oil properly when tested in different laboratories.

The Oil Receptacle.—In the life-test procedure, a 600-ml. beaker was used as the oil container during oxidation, 10-ml. samples of oil being removed for observation at intervals during the run. This procedure was satisfactory for such a test since only minute traces of sludge were present and representative samples of the oxidizing oil could therefore be easily obtained. With sludge-accumulation tests, however, the oil-sampling problem presented a serious difficulty which was solved only by the adoption of individual oil-sample containers. The container adopted was of pyrex glass, 50 ml. capacity, rimless and measured 6 in. in height and 1 in. in diameter. Five such tubes containing 25 ml. of the oil to be tested were immersed in a heavy mineral oil contained in a 600-ml. pyrex glass beaker which was placed on the beaker holder of the life-test oven. The heavy mineral oil (150 sec. Saybolt viscosity at 210 F. (99 C.) and 500 F. (260 C.) minimum flash point) served as an oil bath for the maintenance of the proper oil testing temperature.

In the adoption of the individual oil-sample containers it was recognized that a marked change had been made in the oil depth to surface ratio as compared to the previously used beaker arrangement. From the increased sludging resulting when the oil samples were heated from the container bottom, thereby promoting oil circulation, it might be expected that a marked change in the oil depth to surface ratio would give marked effect on sludging. Such, however, was not the case. Only negligible effect was obtained either in the first trace of sludge formation or in sludge accumulation, Fig. 7. The data of Fig. 7 are obtained on a typical American transformer oil. The oil containers used were tubes 6 in. in length, having an internal area of 7 sq. cm. In one case the oil depth was 3.5 cm. In the other case the oil depth was 8 cm. Two laboratories participated in the investigation.

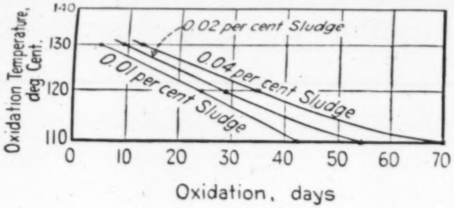

FIG. 4.—Illustrating the Effect of Temperature on the Time to Produce a Definite Amount of Sludge Precipitation

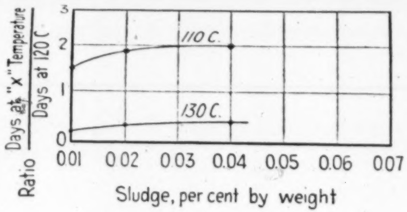

FIG. 5.—Illustrating the Sludging of Transformer Oil as a Function of Temperature Change.

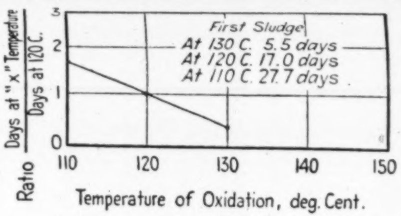

FIG. 6.—Effect of Temperature on the First Traces of Sludge Formation in Mineral Transformer Oil.

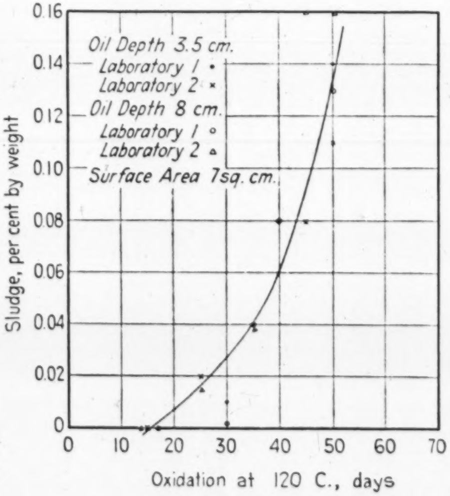

FIG. 7.—Sludge Formation at 120 C. Under the Conditions Prescribed as Independent of the Ratio of Oil Surface to Oil Depth Within the Limits Investigated.

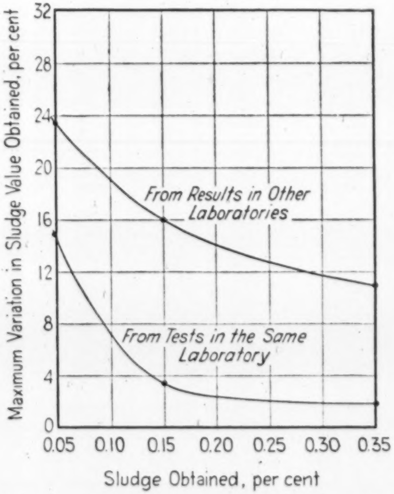

FIG. 8.—Showing the Maximum Variation Obtained in Sludge Measurements Made in Accordance with the Procedure Adopted.

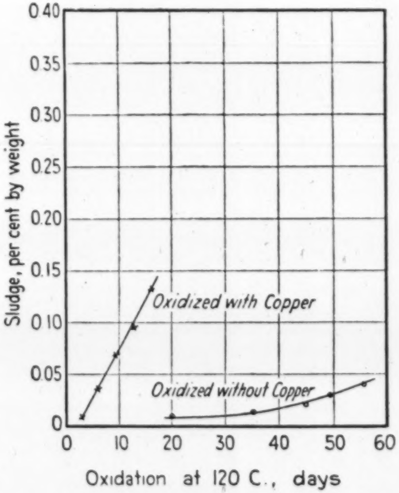

FIG. 9.—Effect of Metallic Copper in Promoting Sludge Formation in Mineral Transformer Oil.

Rate and Conditioning of the Oxygen Supply.—The usual precautions recognized for the life-test procedure were adopted. Air without previous conditioning was passed through a telltale washing bottle containing new transformer oil. The air was passed through at a rate between 1½ cu. ft. per hr. and 2 cu. ft. per hr. The air was then led into the testing oven, being warmed in its passing by contact with the heating element of the oven and was caused to flow over the oil-containing receptacle. Variation in the rate of air supply from 1 to 3 cu. ft. per hr. produced only negligible variation in the sludge-precipitation value.

Method of Sludge Determination.—After prescribed intervals of oxidation, one of the tubes containing the oil under examination was removed from the testing oven and allowed to stand for 24 hr. at room temperature, protected from light and possible contamination. The oil was then diluted with an amount of naphtha equal to 75 to 80 per cent of the oil volume. The naphtha used was according to the requirements for naphtha given in the A.S.T.M. Standard Method of Test for Precipitation Number of Lubricating Oils (D 91 - 35).[4] The naphtha-oil solution was then centrifuged at 1800 r.p.m. for 20 min. The supernatant liquid was carefully decanted and the sludge washed at least twice with naphtha or until no stain was produced on filter paper by a drop of the naphtha washing. The tube with its sludge, after having been properly washed, was dried in a 100 C. oven, the tube being inclined at a 10-deg. angle from horizontal. The tube, after having been cooled in a desiccator, was weighed with the usual gravimetric analysis precautions, freed from sludge by cleaning with acetone or other suitable volatile solvent or cleaning solution, dried at 100 C. and reweighed in order to obtain the total weight of the sludge formed. The weight of sludge formed was expressed in per cent by weight of the original oil.

Using the sludge-weighing procedure outlined, satisfactory test agreement was obtained in determinations made independently in different laboratories. Three samples of sludge-containing transformer oil were examined in each of the eleven cooperating laboratories. As expected, the variation in test results increases as the quantity of sludge in the sample decreases. The results are shown in Fig. 8. From Fig. 8, it is concluded that within the range of sludge formation normally encountered in the examination of mineral transformer oils, the variation in test results traceable to the gravimetric procedure adopted should not exceed 15 per cent for tests in the same laboratory, or 25 per cent for tests in different laboratories. For the larger amounts of sludge present, the allowable variation is materially reduced. Such variation is considered satisfactory for the present stage of the test development. Improvements are expected, however, with the introduction of the usual gravimetric type of analysis at present under consideration. This involves a procedure in which the sludge is actually transferred to and washed on a Gooch crucible before being dried and weighed.

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[4] 1935 Supplement to Book of A.S.T.M. Standards, p. 186.

Use of an Oxidation Accelerator.—The advisability of incorporating an oxidation catalyst as an integral part of a test for gaging sludge formation in transformer oils is of difficult determination. Metallic copper has been frequently suggested for this purpose on the basis that the presence of the copper conductor in the transformer design cannot be ignored in its effect on sludge formation. Copper does act as an accelerator of sludge formation in the usual transformer oil. This is illustrated in Fig. 9 which represents the composite results obtained by eleven cooperating laboratories testing the same oil, first, in the presence of copper and second, in the absence of copper. The fact that copper may affect the sludging of one oil to a greater extent than another, although both are of the usual transformer grade, is discussed in later paragraphs.

In view of the recognized accelerating effect of copper on sludge formation and because of the presence of copper as an inescapable feature of transformer design, it has seemed advisable to include a study of copper effects. The experimental investigation of sludge formation in mineral oil has therefore been divided into two distinct sections. The first deals with the study of a test procedure in the absence of any foreign accelerating material. The second deals with a test procedure in which a copper catalyst is included.

SLUDGE FORMATION STUDIES IN THE ABSENCE OF SLUDGE ACCELERATORS

In the examination of mineral transformer oils in the absence of materials of sludge-acceleration effect, four oils were used. These oils included a commercial type paraffin-base transformer oil (designated as oil C) and three naphthenic base transformer oils, two of which (oils A and B) represented the type of oil customarily used in American transformer practice and one of which (oil D) represented the same base oil but intentionally "under-refined" and considered unsuited for commercial use in the light of past experience. These oils possessed the following characteristics:

	OIL A	OIL B	OIL C	OIL D
Lovibond color	0.10	0.12	0.32	1.80
Saybolt universal viscosity at 38 C., sec	54.4	53.8	51.0	53.4
Condition	clear	clear	clear	clear
Gravity at 15.5/15.5 C. (A. P. I.)	28.3	27.3	36.1	27.2
Acidity	neutral	neutral	neutral	neutral
Flash point, deg. Cent	135	135	154	135
Pour point, deg. Cent	−51	−51	−12	−51

Figure 10 illustrates the average sludge formation characteristics of the oils as originally examined. These results include the data reported by all the cooperating laboratories. The average results given constitute the basis of the critical analysis set forth in subsequent paragraphs.

Differentiation in Sludge Formation Characteristics:

It has already been observed that differentiation between various transformer mineral oils based on the first trace of sludge formation is not always possible due to differences in the rate of sludge deposition in later stages of the oxidation. In like manner, it is impossible to differentiate clearly between transformer oils on the basis of the amount of sludge thrown down in the oxidation interval immediately following the first appearance of sludge, unless the oil tested is of decidedly an inferior quality. Thus in the data of Fig 10 it is evident on the 25-day test that oil D is of a distinctly inferior grade and susceptible to rapid sludge formation. A distinction between oils A, B and C, however, is possible only in the later stages of the oxidation, periods of 45 days or more being required. Whether even then a distinction between the oils is justified from the standpoint of commercial service may well be doubted. Final decision must await examination of the oils in commercial service, a step which has been postponed pending the conclusion of the preliminary laboratory work incidental to setting up a reproducible and logical testing procedure. From the facts available, it is concluded that the sludge examination in accordance with the principles laid down should extend over a sufficient time to produce at least 0.05 per cent sludge by weight.

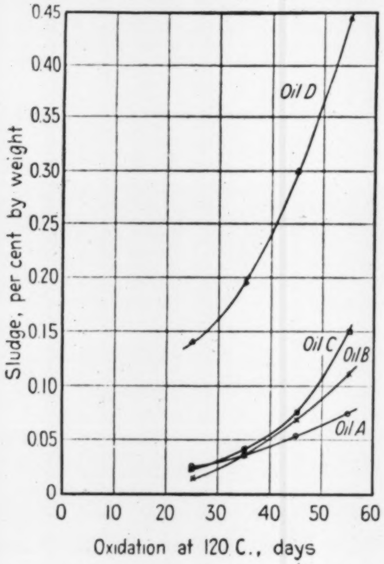

FIG. 10.—The Comparative Sludging Characteristics of the Mineral Oils.

The data are the average values based on the reports from eleven cooperating laboratories.

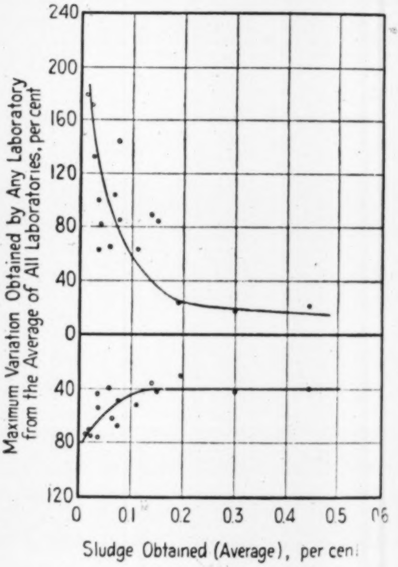

FIG. 11.—Showing the Maximum Variation in Sludge Results Obtained for Any Testing Laboratory as a Function of the Amount of Sludge Under Examination.

Agreement Between Testing Laboratories:

In examining the data submitted by the eleven cooperating laboratories, the agreement between the results reported has been studied from two standpoints. The examination has been made, first, as to the relative sludge-forming characteristics of the four oils and, second, as to the variation in sludge formation reported for each oil.

With respect to the relative sludge-formation values of the four oils, the data are summarized in Table I. In this table the oils are ranked in accordance with their increasing sludge tendency. The listing A-B-C-D therefore indicates that oil A has the least sludging and oil D the greatest sludging tendency. Analysis of the data of Table I indicates the necessity of extending the oil oxidation for a period well beyond the initial stages of the sludge formation. Different laboratories would rank the relative sludging propensity of the oil differently, depending upon the degree of oxidation. For the longer periods of oxidation, greater agreement between the laboratories is obtained. As indicated in Fig. 10, the average results show that the oils should be classed as A-B-C-D, oil A being least sludging and oil D being most sludging.

From the analysis of the sludging data in such a manner, it is concluded that although all the laboratories are able to detect clearly the distinction between oils of high and low sludging tendencies, the test as set up will not allow complete agreement in rating oils which might be accepted as falling in the group characterized by good sludge resistance. Difficulty in rating the oils shows up even in tests carried out at different times in the same laboratory by the same operator and with the same equipment. A typical result is given in Table II.

Viewed from the standpoint of actual per cent sludge values reported, the variation of each laboratory from the average of all test results depends both on the quantity of sludge obtained and on the individual variation peculiar to each testing laboratory. Figure 11 summarizes the maximum per cent variation obtained for any laboratory from the grand average of all laboratories as a function of the amount of sludge under examination.

It is recognized that the maximum percentage variation from the average per cent sludge value obtained by the eleven cooperating laboratories is too great. Efforts at present are being directed toward the reduction of the maximum allowable variation in sludge results, especially in the analysis of sludge present in quantities less than 0.1 per cent.

Despite the fact that the variation between test results depends on the amount of sludge under examination, it is obvious that part at least of the discrepancy is dependent on the variations in those factors underlying the actual oil oxidation. This is shown by the analysis of the testing results from each laboratory. In Table III, the total results reported by each laboratory for each oil for all testing periods of oxidation are averaged

TABLE I.—THE COMPARATIVE SLUDGE RATING OF OILS BY EACH COOPERATING LABORATORY.

Laboratory	Oxidation at 120 C.			
	25 days	35 days	45 days	55 days
No. 1	B–A–C–D	B–C–A–D	....	A–B–C–D
No. 2	B–C–A–D	B–C–A–D	C–B–A–D	B–C–A–D
No. 3	A–B–C–D	A–B–C–D	A–B–C–D	A–B–C–D
No. 4	C–A–B–D	A–C–B–D	....	....
No. 5	....	B–C–A–D	B–A–C–D	A–B–C–D
No. 6	A–B–C–D	A–B–C–D	A–C–B–D	A–C–B–D
No. 7	A–C–B–D	A–C–B–D	A–B–C–D	A–B–C–D
No. 8	....	C–A–B–D	C–A–B–D	A–B–C–D
No. 9	A–B–C–D	B–A–C–D	A–B–C–D	C–A–B–D
No. 10	A–B–C–D	....	A–B–C–D	A–B–C–D
No. 11	C–A–B–D	B–A–C–D	A–B–C–D	A–B–C–D

TABLE II.—OIL SLUDGE RATING ON BASIS OF CHECK RUNS MADE IN SAME LABORATORY.

Laboratory		Oxidation at 120 C.			
		25 days	35 days	45 days	55 days
No. 6	Run 1	A–B–C–D	A–B–C–D	A–C–B–D	A–C–B–D
	Run 2	A–C–B–D	A–C–B–D	A–B–C–D	A–B–C–D
No. 11	Run 1	A–B–C–D	B–A–C–D	A–B–C–D	C–A–B–D
	Run 2	C–A–B–D	B–A–C–D	A–B–D–C	A–B–C–D

TABLE III.—INDIVIDUAL LABORATORY VARIATION IN SLUDGE VALUES FROM GRAND AVERAGE BASED ON DATA FROM ALL LABORATORIES.

Laboratory	Average Deviation from Grand Average, per cent				Average of Oils A-B-C-D
	Oil A	Oil B	Oil C	Oil D	
No. 1	+106	+101	+120	+35	+90
No. 2	+9	–25	–29	–41	–26
No. 3	–43	–39	–47	–35	–41
No. 4	–41	–30	–28	–18	–29
No. 5	–47	–35	–42	–11	–34
No. 6	–14	+26	+17	+6	+9
No. 7	+52	–14	+28	–7	+15
No. 8	–57	–58	–62	+11	–41
No. 9	–23	–26	–8	+33	–6
No. 10	–4	+2	+5	+1	+1
No. 11	+5	+105	+56	+53	+66

TABLE IV.—VARIATION IN SLUDGE RESULTS REPORTED BY LABORATORY NO. 1 WHEN SAME OIL IS TESTED FOR SLUDGE FORMATION AT DIFFERENT PERIODS OF TIME BUT WITH SAME TESTING APPARATUS AND OPERATOR.

NOTE.—Figures in parentheses are data obtained in the check run.

Days at 120 C.	Sludge Formed, per cent by weight			
	Oil A	Oil B	Oil C	Oil D
21	0.0018 (0.0111)	0.0018 (0.0088)	0.0000 (0.0093)	0.0574 (0.0879)
35	0.0079 (0.0135)	0.0196 (0.0176)	0.0076 (0.0194)	0.1663 (0.1212)
42	0.0213 (0.0230)	0.0231 (0.0294)	0.0243 (0.0215)	0.1715 (0.1760)
49	0.0298 (0.0112)	0.0431 (0.0281)	0.0242 (0.0114)	0.2565 (0.2355)
56	0.0442 (0.0363)	0.0575 (0.0531)	0.0398 (0.0392)	0.3295 (0.3235)

and expressed as percentage variation from the total average sludge formed. It will be observed that in almost every case a laboratory reporting high (or low) sludge values for one oil invariably reported correspondingly high (or low) sludge values for all oils.

In Table IV are given the sludge values reported for two oxidation runs taken on oils A, B, C and D in laboratory No. 1, the runs being carried out successively under as nearly duplicate conditions as possible.

Reproducibility of Average Sludge Values:

To demonstrate further the degree of reproducibility of the sludge-accumulation type of test, the cooperating laboratories again examined the sludging characteristics of three of the oils one year after the completion of the first series of tests described in the foregoing paragraphs. Duplicate samples of oils A, B and D from the same manufacturing batches of the previous year and identical therewith were distributed for this purpose. The testing results with respect to the individual variations and vagaries were similar to the results of the previous tests. For the clear differentiation between oils, it was again concluded that the oxidation period should extend at least long enough to produce 0.05 per cent sludge by weight. The relative classification of the three oils was in agreement with the work of the previous year. Perhaps the most striking result was the parallelism in the average sludge values reported for each year. Illustrative of the agreement between the average sludge results for each oil, the following data for the 55 to 56-day period of oxidation are cited:

	Oxidation at 120 C., days	Sludge, per cent by weight		
		Oil A	Oil B	Oil D
Original test average (11 laboratories)	55	0.075	0.110	0.445
Second year test average (11 laboratories)	56	0.075	0.104	0.443

The close agreement in the average sludge value for each of the three oils obtained over a 2-yr. testing period is noteworthy in indicating that the general type of test under investigation is reproducible. It is to be observed, however, that the close agreement given covered an oxidation period of sufficient length that the amount of sludge formed was in excess of 0.05 per cent.

It is of further interest to observe that in accordance with Fig. 5, the effect of temperature change on sludge formation in mineral transformer oil approaches the normally-expected value when the amount of sludge obtained is of the order of 0.05 per cent. Thus for small amounts of sludge (0.01 per cent) when the temperature of oxidation is reduced from 120 to 110 C., the time to produce 0.01 per cent sludge is increased only 50 per cent. To produce 0.04 per cent sludge, however, the oxidation period is doubled when the temperature is changed from 120 to 110 C. On the other hand, when the oxidation temperature is changed from 120 to 130 C., the time to produce 0.01 per cent sludge is reduced to as low as 20 per cent of the 120 C. time interval. To produce 0.04 per cent sludge, the time interval is reduced only to 40 per cent of the 120 C. time interval. Since one would expect that changing the temperature of oxidation 10 deg. Cent. would approximately double the sludge-formation rate for an increase in temperature and halve the sludge-formation rate for a decrease in temperature, it is to be concluded that an abnormal condition exists in the early stages of the oxidation run which tends to produce sludge at a faster rate than would be expected. It is in this range of sludge formation that the greatest variation in test results is obtained.

SLUDGE FORMATION IN THE PRESENCE OF A SLUDGE ACCELERATOR

The use of an accelerator of oxidation in the sludge testing of transformer oils is recognized as a possible source of unexplainable variation in the test results. Copper, because of its general use in transformer design, has heretofore been the commonly selected accelerator of sludge formation, and was so selected in the examination of the transformer oils herein described. It is not overlooked, however, that the formation of copper oxide on the surface of the metallic copper during the oxidation run may be of effect on the test results. Figure 12 illustrates the behavior observed for a typical American transformer oil tested without an accelerator at 120 C. and at 90 C. with copper and with copper (cupric) oxide. The marked acceleration in sludge accumulation for the oil sample in contact with the copper oxide is to be noted. The data presented are limited and the greater effect of the copper oxide as compared to the effect of the copper may be due to its greater surface area. Future investigation will include a detailed study of the comparative effect of copper and copper oxide as accelerators of sludge formation.

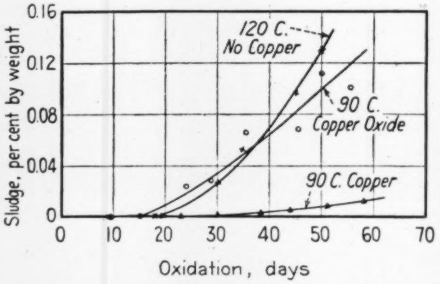

FIG. 12.—Effect of Copper and Copper (Cupric) Oxide on Sludge Formation in a Typical American Transformer Oil.

Testing Technique with a Copper Accelerator:

The testing procedure for the oxidation of transformer oil in the presence of copper duplicates that already described in previous paragraphs for tests in the absence of copper. The copper accelerator was in the form of wire 0.040 in. in diameter and 12 in. long, the surface of which was carefully cleaned with a fine abrasive paper. The wire was wound on a $\frac{1}{2}$-in. mandrel to a spiral length of 2 in., washed with ethyl ether and dried.

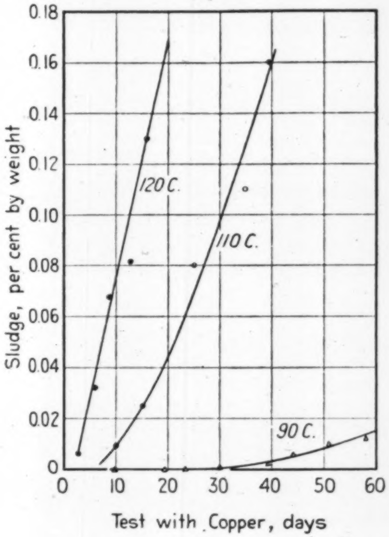

FIG. 13.—Effect of Copper on Sludge Formation in Transformer Oil as a Function of the Testing Temperature.

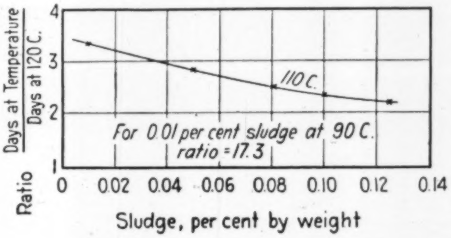

FIG. 14.—Illustrating the Effect of Temperature on the Rate of Sludge Precipitation as a Function of the Oxidation Period.

In the examination of the oxidized oil for sludge content, the testing procedure is exactly as followed for the no-copper test, modified as is necessary due to the presence of copper. After having been cooled to room temperature, and held for 24 hr., the copper spiral is removed from the oxidized oil and washed with a spray of the specified naphtha. The oil is then diluted with naphtha to 75 to 80 per cent of its volume and centrifuged, the sludge washed at least twice with naphtha with intervening centrifuging, and the separated sludge is dried at 100 C. The amount of sludge is expressed in per cent by weight of the original oil used.

The oils tested were selected to include wide variations in the refining treatment and were designated as oils A, B, C and D. These oils were identical with the corresponding oils of like designation already described under the no-copper testing procedure.

Effect of Temperature:

From the shape of the sludge-time curve of Fig. 13, it is obvious that the effect of changing temperature on sludge formation in transformer oil in the presence of copper differs materially from the effect of temperature on the same oil in the absence of copper. This is clearly brought out in a comparison of Figs. 14 and 5. Whereas in the absence of copper the formation of sludge during the early stages of oxidation of the oil at 110 C. was at a faster rate than would be normally expected from the 120 C. test, the same oil in the presence of copper shows sludge formation at a slower rate than would be expected for the 110 C. test. Stated otherwise, in the absence of copper the time necessary to produce 0.01 per cent sludge at 110 C. was only 50 per cent longer than the time to produce the same amount of sludge at 120 C. In the presence of copper, however, the time to produce 0.01 per cent sludge was 3.3 times as long at 110 C. than at 120 C. However, just as in the absence of copper the sludging rate approaches the normally-expected value as the amount of sludge is allowed to accumulate, so too at 110 C. in the presence of copper the rate of sludge formation approaches the normally-expected value as the test is extended. Thus the test to produce 0.125 per cent sludge at 110 C. in the presence of copper is approximately 2.2 times the duration of oxidation necessary to produce the same amount of sludge at 120 C.

It is obvious that the difficulties of controlling the oxidation of the oil increase with the extension of the test. It is further desirable to oxidize the oil at as low a temperature as possible consistent with the desire not to unduly prolong the oxidation. In the light of the results illustrated in Figs. 13 and 14, the temperature of 120 C. was arbitrarily accepted and the time interval of oxidation limited to not more than 25 days. With the type of oil customarily used in American transformer practice, this would result in approximately 0.20 per cent sludge by weight. From the data obtained, it is concluded that the temperature of 120 C. adopted for the sludge test does not introduce any abnormalities in the sludge-formation reactions.

Amount of Sludge Retained on the Copper Wire:

The copper wire after oil oxidation was merely washed with naphtha. This treatment has been found satisfactory since the sludge retained on the copper is negligible in amount except possibly for tests of long duration. Typical data illustrative of the amount of sludge retained on the copper during the oxidation run are indicated in Fig. 15. The data of Fig. 15 represent averages of the total data reported by the eleven cooperating laboratories. From Fig. 15 it is concluded that even for tests lasting as long as 16 days, not more than 5 per cent of the total amount of sludge formed is retained on the copper surface. For tests of shorter duration (less than 9 days), no weighable amount of sludge is retained on the copper surface.

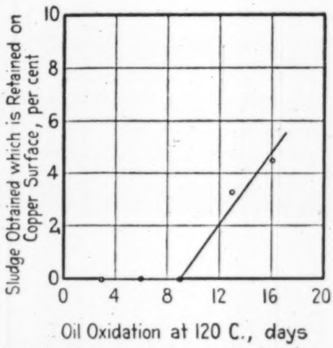

FIG. 15.—Amount of the Total Sludge Formed Which Is Retained on the Copper Surface During the Oil Oxidation Run.

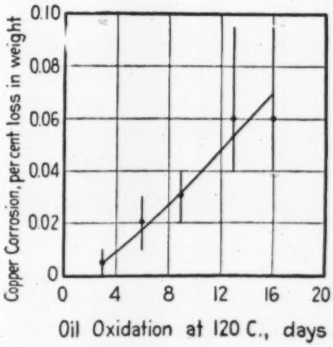

FIG. 16.—Corrosion of the Copper Catalyst During the 120 C. Oil Oxidation Test.

Chemical Corrosion of the Copper Surface:

Aside from a slight darkening of the copper surface in some instances, no evidence of copper corrosion during the oxidation run was encountered. The weight change of the copper during the test was negligible. While in some instances the copper decreased slightly in weight, the usual trend showed a slight increase. Figure 16 illustrates the average copper corrosion reported by the eleven cooperating laboratories, showing as well the maximum and minimum corrosion figures obtained.

The slight but detectable change in the copper surface varying to some extent from run to run should not be overlooked. It may indicate a change to which is attributable the variation in the sludge-forming characteristics of the oils reported by the cooperating laboratories.

Sludge-Accumulation Characteristics in the Presence of Copper:

The presence of copper, as expected, accelerated the sludge formation. The average of all sludge results reported by the eleven cooperating laboratories is illustrated for each of the oils in Fig. 17. From the data of Fig. 17, it is to be observed that many of the findings already described for the no-copper oxidation test apply with equal force to the test in the presence of copper. Just as it was impossible to distinguish clearly between the oils on the basis of the first sludge formation or even in the interval immediately following the first appearance of sludge, so it is necessary with copper to withhold a decision until sludge formation has reached significant proportions. For example, on the third day with copper, oil D of known inferiority shows almost to equal advantage with the other oils. On the sixth day oil D showed clearly as a rapidly sludging material. On the sixth day, however, no distinction can be drawn between oils A and B. Even when the sludge has accumulated to approximately 0.10 per cent, these oils appear equivalent. They allow differentiation after extreme oxidation and then only to an extent which makes the decision of doubtful value. Oil C, however, is clearly distinguished as being characterized by intermediate sludge tendencies.

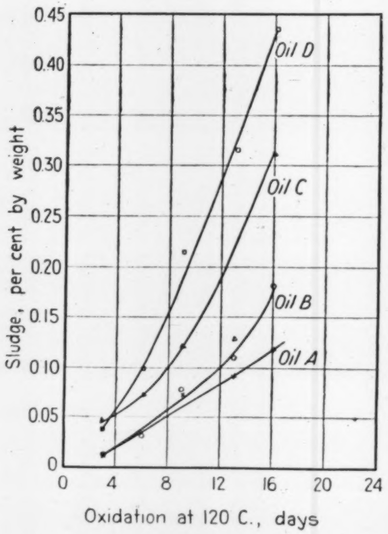

FIG. 17.—Average Sludge Forming Characteristics of the Oils Tested in the Presence of Copper.

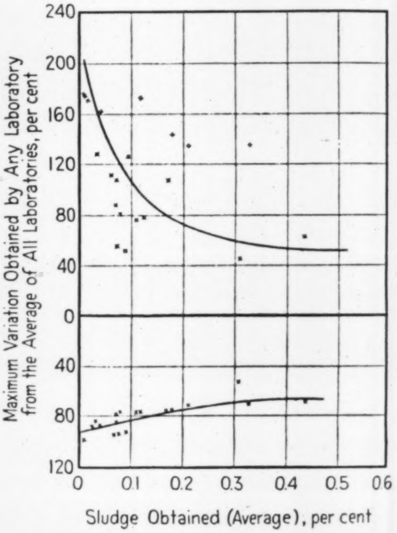

FIG. 18.—Maximum Variation in Sludge Values Obtained for Oxidation Tests Run in Different Laboratories in the Presence of Copper.

From the data presented in both the no-copper and the copper tests, it is concluded that to distinguish between oils, with the type of oxidation procedure used, it is necessary to obtain an amount of sludge in excess of 0.05 per cent. Without copper, this demands with the grade of oil in American transformer use at least as long as 35 to 45 days at 120 C. With copper, a period of oxidation at least as long as about 9 days is needed.

Although the no-copper and the copper test results eventually rate the oils in the order oil A (least sludging), oil B, oil C and oil D (most sludging) there is another effect of copper which must be observed in addition to its acceleration of the sludge-forming characteristics. It has already been observed that copper will vary in its effect on sludge formation in different oils. This is clearly brought out in the test data submitted for oils A, B, C and D. The effect of copper is greatest on those oils of the normal transformer type which have the greatest sludging resistance without copper. This is illustrated in comparing Figs. 10 and 17 which show the average sludging properties of oils A and D, two oils of naphthenic base differing only in the degree of refining treatment. Oil A possesses acknowledgedly less tendency to sludge formation than oil D. After 25 days of oxidation without copper at 120 C., oil A gave 0.025 per cent sludge, an amount exactly equal to the sludge formed in the same oil after only $4\frac{1}{2}$ days test in the presence of copper. Oil D, however, gave only 50 per cent as much sludge after $4\frac{1}{2}$ days with copper as compared to the amount deposited after 25 days without copper. Or again, take the condition of extreme oxidation. After 55 days test without copper, oil D gave 0.445 per cent sludge, an amount almost identical with the sludge formed (0.435 per cent) after 16 days test at the same temperature of 120 C. with copper. During the same intervals of oxidation, however, oil A showed 60 per cent more sludge formation due to the presence of copper, depositing 0.075 per cent sludge at 120 C. in the absence of copper and 0.120 per cent sludge in its presence. From such behavior, it can only be concluded that the incorporation of copper in the sludge test may tend to eliminate the difference in the comparative sludge formation in oils clearly evident when oxidized without copper.

Reliability of Sludge Tests with Copper:

Figure 18 illustrates the maximum percentage variation in test results from the average of all cooperating laboratory data. The percentage variation is reported as a function of the amount of sludge formed.

In general, it is to be observed that the variation in sludge values reported by the various laboratories tends to decrease as the quantity of sludge measured increases. The decrease in sludge variation, however, is not so pronounced as already illustrated for tests without copper. In comparing the test results, it must be remembered that the amount of sludge formed in the presence of copper is generally greater than the quantity of sludge examined in the test without copper.

Use of Conditioned Air:

In an attempt to reduce the test variation, oils A, B and D were again tested by the cooperating laboratories. The test procedure was exactly as before with the exception that the air supply producing the oxidation was conditioned before entrance to the oxidizing oven. The conditioning was accomplished by passing the air through a drying tube (Eimer & Amend No. 22556) containing Dehydrite (magnesium perchlorate trihydrate), changed weekly. The air was then passed through a gas-bubbling bottle containing unoxidized transformer oil, changed weekly. The gas-washing bottle was of the Eimer & Amend type No. 18842 with 500 ml. capacity.

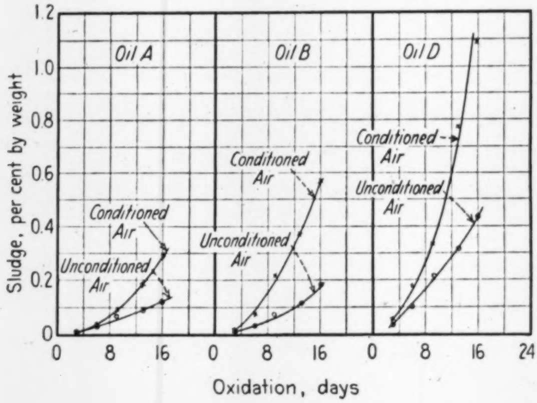

FIG. 19.—Effect of Conditioned Air on the Sludging of Mineral Oil in the Presence of Copper.

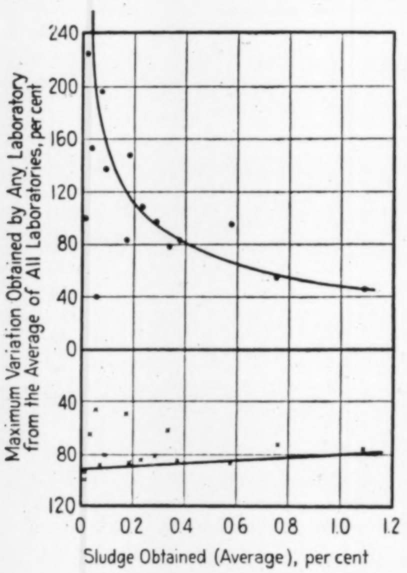

FIG. 20.—Maximum Variation in Sludge Values Obtained for Oxidation Tests Run in Different Laboratories in the Presence of Copper Using Conditioned Air.

Using conditioned air, each laboratory reported increased sludge formation in the oils examined. The average results for all the tests reported are illustrated in Fig. 19, in which comparison is made with the sludge data reported for unconditioned air.

Figure 20 shows the maximum variation from the grand average of all sludge results obtained using conditioned air, expressed as a function of the quantity of sludge obtained. Again the percentage variation decreases as the amount of sludge formed increases. In general, it appears that the selection of conditioned or unconditioned air has but little influence on the variation in the test results obtained. It appears that other factors of the oxidation such as the temperature variation and differences in the copper surface may be largely responsible for the difficulty in obtaining closely reproducible results in a test of this character.

SUMMARY

The sludge-accumulation test appears capable of distinguishing between oils of varying sludging quality only if carried sufficiently far to produce at least as much as 0.05 per cent sludge by weight. This applies whether or not copper be used as an accelerator of the oxidation. Without copper, the test appears more easily reproduced but must be extended for at least 35 to 45 days at 120 C. under the conditions outlined. With copper, a decision in as short a time as 9 days at 120 C. is possible with the oil of a quality approaching the average sludge resistance of the usual American transformer oil. With oil definitely of an inferior quality, both tests are capable of application in even a shorter oxidation time. Not all oils appear affected to a like degree in contact with copper during the oxidation. In general, the oils of the usual transformer type are more greatly accelerated by copper than are the oils considered to be of less sludge resistance in the absence of copper. The result is that the presence of copper may so modify the sludging reactions as to mask the differences clearly evident when the same oils are sludged in the absence of copper. The significance of this fact can only be gaged when the investigation reaches that stage when the oils will be examined from the standpoint of actual transformer service.

The variation in sludge results obtained in different laboratories, even with the larger amounts of sludge formed, are of such magnitude that the sludge-accumulation test is at present not considered as satisfactory for standardization. Those factors which affect the sludging of an oil and which are responsible for the variation in sludge results reported by different laboratories are being investigated and eliminated. Progress has been definitely made as a result of the cooperative work of the various laboratories represented in these activities. Continued progress may be expected to the end that a well-grounded test, capable of giving reproducible results and definitely related to the actual commercial use of transformer oils, will be established.

DISCUSSION

MR. HANS STÄGER[1] (*by letter*).—This paper discusses the sludge-forming tendency as a requisite for a good transformer oil. While the sludge is one among several other possible products of the reaction, and has a certain significance, it is by no means the determining one. Far more important, in our opinion, are the products of oxidation by air at higher temperature, which are not precipitated as sludge and have the character of peroxides. Such products are very dangerous for the reason that they destroy the organic insulation materials of the transformer within a short time. The cellulose is transformed to oxycellulose, which is a white powder and therefore has no more mechanical strength and consistency, and in addition to that has lost its electrical insulation qualities. Unfortunately it is those very oils which do not form any sludge in service which have the tendency to decompose in peroxide products by which the insulation is endangered to a high degree. A method of testing which considers only the formation of sludge is, therefore, fundamentally deficient.

As to the formation of sludge proper, it may be said that the catalytic influence of copper is known as mentioned in *Industrial and Engineering Chemistry* in 1925.[2] The characteristic of the reaction is not in any way changed by the presence of copper. In other words, the resulting reaction products are identical in all respects with those which would form without copper. The only effect is that the speed of the reaction is increased by the addition of the copper.

The temperature has a determining influence on the reaction. Several investigators have shown that the critical temperature is at about 115 to 120 C. depending on the type and degree of refining treatment of the crude oil. Below the critical temperatures the reaction proceeds in the manner of a monomolecular reaction and above that temperature it changes to a bimolecular reaction, which is a fundamentally different one. A temperature of 120 C. should, therefore, not be chosen for the test, as one then incurs the danger of operating in the region of the bimolecular reaction.

A test intended to simulate the aging of transformer oils in service should not be based upon the formation of sludge alone. At the start of the oxidation no sludge is formed, but soluble acid reaction products from which the insoluble sludge is derived by formation of anhydride, condensation and polymerization by the way of soluble sludge. This insoluble sludge alone is taken into consideration by the above-mentioned test. There are oils which form considerable quantities of sludge, which may become very dangerous in the cooling systems.

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[1] Chemist, Privatdozent an der Eidg. Technischen Hochschule, Zurich, Switzerland.

[2] Hans C. Stäger, "Method of Testing Transformer Oils," *Industrial and Engineering Chemistry*, Vol. XVII, No. 12, pp. 1272–1275 (1925).

The acid reaction products, which, depending on their properties, may become very dangerous, are not determined in the method under discussion. The same is true of the extremely dangerous, neutral oxidation products of the peroxide character—these being the most important destroyers of the organic and cellulose insulating materials.

Mineral oils tested with the method may be found suitable for service in transformers, and yet have all the bad properties possible, because this method is based exclusively upon one single reaction product and does not consider the others, which are by far more important.

MESSRS. HENRI WEISS[3] AND T. SALOMON[3] (*by letter*[4]).—We have been successful in checking some of the results presented in this paper in spite of the differences in apparatus and method used for the determination of sludge formed. This convinces us that it should be possible to obtain, by judicious collaboration, an agreement with a view to formulating an international method for the purpose of estimating the relative aging tendencies of new transformer oils.

It is noteworthy that the sludge curves are parabolic; this checks with our results and those of Mr. Stäger. The curves differ in their angle of inclination, some being flat and others rising rapidly; as a result of our tests, this characteristic of curves is less related to the quality of the oil than to its degree of "purity." We have, in addition, discovered that some oils, moderately refined but still containing some traces of products of the reaction with sulfuric acid, show a more rapid acceleration of the speed of sludge formation (shown by sharp rise of the sludge curve) than others of the same quality, but, carefully freed of the last traces of the reaction, they form very flat curves. Refiners have never given sufficient attention to the problem of the "purification" of oil treated with sulfuric acid by washing with alkali, water, etc., the problem is at least as important as the refining with sulfuric acid itself.

We are now preparing methods for determining the degree of purity of oil, but these methods are not yet generally applicable. The problem is, however, important because the presence of these "impurities of refining" influences the behavior of oils in service.

It seems to us that the important deviations which have been noted in the amount of sludge obtained in the same laboratory or in different laboratories are explained in a large measure by the incomplete precipitation of sludge obtained with the technique employed. From our own tests, which have been checked by Mr. Woog, it is essential to dilute the oil with at least three volumes of standard gasoline and to allow the mixture to stand from 16 to 24 hr. protected from sunlight, in order to obtain a precipitation not only complete but of constant value; moreover, this is verified by replacing volumetric determinations of sludge formation by gravimetric determinations as pointed out in the report. For the filtration of sludge we successfully used Schott (Jéna) 1G4 crucibles of fritted glass which are more convenient to use than the Gooch crucibles with asbestos. With the concentration used by the authors, they obtained a partial and incomplete precipitation, the amount of which varied from one test to another.

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[4] Translated by E. A. Snyder.

We are convinced that, by using three parts of standard gasoline to one part of oil, results are obtained that are higher perhaps, but also more constant. This technique appears to be inapplicable for certain oils containing chemical impurities, such as traces of corrosive sulfur; in such cases variable results are always obtained. In order to obtain constant results, it is necessary, according to the results of our studies, to extract the aged oil with acetone, to evaporate the acetonic extract and to precipitate the sludge by adding three times as much standard gasoline as oil used in the test. The precipitation is instantaneous and the precipitated sludge can be filtered at once.

The catalytic effect of copper is not the same for all qualities of oil. The authors' tests agree perfectly with ours in the sense that the catalytic effect of copper is slight for oils of inferior quality and much greater for oils of very good quality. We have also discovered some oils on which the catalytic action of the copper is negligible.

This variation in the action of copper is explained by the fact that the metal serves only to start the aging; an oil of poor quality contains other products capable of producing the same effect; the function of the copper is thus reduced. For oils of very good quality, on the other hand, that contain little or none of these "starter" products, the action of the copper is greater. In certain exceptional cases, the action of the copper can be partially or completely neutralized by the fact that the surface of the copper is covered with a sheath of corrosion products by the oil, preventing its contact with the copper.

We have, however, never encountered any cases of new oil, the relative classification of which would have been changed as a result of the presence of copper in the aging test.

Our own tests show that the formation of sludge is a very complex reaction and that it is necessary to follow the complete process of this phenomenon in order to obtain a rational classification of oils. Some tests, too limited either in duration or in quantity of sludge formed, could in many cases convey an erroneous impression of the aging tendency of oils.

MESSRS. F. M. CLARK[5] AND E. A. SNYDER[6] (*authors' closure by letter*).—The comments of Messrs. Weiss and Salomon need no reply as they do not contradict or take exception to any of our conclusions. It is quite interesting and encouraging to note that our results and conclusions agree perfectly with those obtained by them, even though they were working with different apparatus and methods. Their comment in regard to possible incomplete precipitation of sludge is noted. Some work on this point has already been carried out by our committee, but it will be further investigated.

Mr. Stäger's comments are interesting, but we believe they are based on the erroneous idea that our paper assumes the sludge-forming characteristics of a transformer oil to be the determining characteristic of quality. Such may or may not be the case, but our paper makes no claims for or against such a theory. Our committee realizes that the sludge-forming characteristics of a transformer oil is at least one of its important characteristics, and in fact so important that practically all specifications, either in this country or abroad, specify some method for its determination.

The members of our committee are also well aware of the fact that no satisfactory method for the determination of this characteristic has ever been formulated and consequently a great deal of its work has been directed towards the development of a sludge test that would be free of objections cited against sludge tests developed heretofore and at the same time be capable of giving reproducible check results in the same and also in different laboratories by different manipulators. Our paper is a summary of the results obtained along this line of investigation. Whether the soluble products of oxidation, the peroxides or so-called acids developed, are more harmful in a transformer than deposited sludge is a question that is entirely a matter of specifications and engineering design and its discussion does not properly belong in the scope of our paper.

Mr. Stäger comments further that the critical temperature of oils has been found to be 115 to 120 C. by certain investigators. It is also fair to state that some investigators have found this critical temperature to be considerably above 120 C. Our committee had this point in mind during the investigation and brief reference is made to it on page 572 of the paper, where we state that, "As seen from Figs. 4 and 5, the variation in testing temperature between 110 and 130 C. produces no abnormality which would be indicative of a marked change in the oil-sludging characteristic." We believe that a study of Figs. 4, 5 and 6 bear out our contention that no critical range was shown up to a temperature of 130 C. One independent investigator in this country, whose findings are not as yet published, stated that in his investigations he had found no critical range in passing from 90 C. to temperatures of 140 to 150 C., but that the reactions appeared to be continuously similar all through that temperature interval.

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[5] Chemist, General Electric Co., Pittsfield, Mass.
[6] Socony-Vacuum Oil Co., Inc., New York City.

SIGNIFICANCE OF MATERIAL TESTS AND OF DESIGN TESTS UPON ELECTRICAL INSULATING MATERIALS

By L. E. Barringer[1]

Synopsis

After outlining the purposes of material tests, and pointing out their very great service in establishing the basic properties of different electrical insulating materials, this paper emphasizes the fact that the properties determined in this manner may not carry over unaltered into all design formations that may be contemplated. Therein lies the reason for a need to recognize that sometimes there may be a marked difference between the results of basic tests on laboratory samples and of performance tests on those same materials after they have been fashioned into the shape required by a particular design. The primary object of this paper is to point out that such differences do exist and to warn the designer to anticipate the possibility of encountering them. That the results of the two types of tests are not always in agreement should not be regarded as casting any discredit on the accuracy of either or on its usefulness; but it does mean that intelligent consideration should be given to weigh the significance which each may have in any specific design problem.

There is not always a clear understanding on the part of buyers and users of electrical insulating materials as to the relative significance and practical application of (*a*) tests made to determine the comparative values of such materials for certain purposes and (*b*) tests made to reveal their actual performance in specific designs.

It is of course often necessary to compare various insulating materials without regard to highly definitive uses, as in research and development work when various types of standard materials are being investigated to establish their suitability for a general field or the same type of material from different sources are being examined to record agreement with generally accepted properties for that type.

For instance, since porcelain is largely used in connection with high-tension transmission lines, outdoor switching equipment, transformers and lightning-arrester apparatus, it must be weatherproof, mechanically strong, resistant to mineral oils or other transformer insulating liquids and of high dielectric value, unchangeable with age.

Weatherproofness includes water insolubility, imperviousness to water (without pores or capillaries to absorb water and thereby cause both reduction of dielectric strength and physical disruption upon freezing and thawing), resistance to high and low atmospheric temperatures within short cycles (as in Arizona), and toughness to resist the impact of hailstones, and static mechanical strength to resist abnormal stresses imposed by wind-blown and sleet-laden conductors. Any so-called porcelain which will not meet the necessary weatherproof requirements satisfactorily is not safe to use, whether it be for a line insulator, transformer bushing, lightning-arrester casing or switch post.

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[1] Engineer of Insulations, General Electric Co., Schenectady, N. Y.

In order that different investigators, working independently, can determine and rate the general suitability of porcelain for such purposes, standard test procedures are necessary since variation in test methods creates difficulties in arriving at a reliable comparison. This importance of standard tests for the evaluation of insulating materials was pointed out by the author in a previous article.[2]

Knowledge of the basic properties of materials, such as is obtained from the testing of specimens, is also desirable and often essential prior to designing in order to secure the most effective use of such materials. For example, a material of which the compressive strength is high but the tensile and cross-breaking strengths comparatively low would naturally be used in designing so as to subject the finished insulator in service to compressive rather than tensile and transverse strains. In certain woods or in molded laminated materials in which the dielectric strength is considerably greater across rather than parallel to the grain or laminae the designer of insulating parts gives due consideration to these relative values in providing effective insulation in conjunction with other functional requirements.

Even the user of varnished cloth tapes might use information concerning dielectric and physical strengths as a basis to determine whether the material should be employed as bias or straight-cut, and in what width and thickness.

In all such cases, information with respect to the specific and relative values of insulating materials is highly valuable to the designer and user. Such information is best procured from the results of carefully devised tests applied to the simplest forms of test specimens, such as those sponsored by the A.S.T.M.

The American Society for Testing Materials has as one of its foremost objectives the devising of reliable methods of test for industrial materials so that intelligent and dependable comparisons can be made. Without such carefully developed methods of test the situation with respect to the evaluation of materials would indeed be chaotic.

RELATION OF DESIGN TO BASIC PROPERTIES

The primary purpose of this article is to emphasize that, though suitable standard laboratory tests reveal differences in the values of the critical properties of a given type of insulating material, these unit values should not be expected to coincide precisely with values derived from the results of applying tests for the same properties to the same material in all forms or designs, particularly in assembled units.

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[2] L. E. Barringer, "The Importance of Standards in the Evaluation of Insulating Materials," *General Electric Review*, Vol. XXVII, No. 11, November, 1924, p. 736.

Again taking porcelain as an example, the dielectric strength of an insulator varies with its thickness, imposed mechanical stresses, its temperature and the shape of assembled metal components. The mechanical strength will vary with "fit of glaze," method of firing and with the shape or design. Dry and wet flashover voltages depend entirely upon contour. Corona phenomena are influenced by both contour and type of glaze. This interdependence of properties has been previously discussed by the author in an article entitled, "The Relation of Chemical and Physical Structure to Dielectric Behavior."[3]

It is evident, therefore, that laboratory tests made upon simple test specimens will seldom coincide with the results of combined mechanical and electrical tests applied to any insulator shape or assembly. When load is applied to an insulator unit, consisting of porcelain and metal components joined with cement or by mechanical means, there is a complexity of strains varying in distribution and intensity with every design and affecting to a correspondingly varying degree the electrical breakdown value under these strain conditions.

Parmelee and Kraehenbuehl[4] have indicated that there is apparently no specific law covering the relationship of electrical breakdown and tensile loads nor any definite or continuous correlation even between the tensile and compressive strengths, although while under a compressive strain there does seem to be a marked and definite law of performance under a simultaneously applied electrical stress.

Since in commercial designs there are very often both compressive and tensile stresses, computations cannot safely be made upon the basis of laboratory tests to predict the performance of any given design under a combined mechanical and electrical test. A design test is essential for the purpose.

Likewise, the results of testing standard specimen forms for resistance to abrupt or extreme changes of temperature cannot be mathematically applied to irregular insulator forms in which adjoining thin and thick sections of differential heat conductivity constitute a condition quite different from that in a test specimen of comparatively uniform cross section. An even-walled casserole of Coors or Meissen porcelain may be thrust suddenly into the flame of a bunsen burner without its cracking, but identically the same porcelain composition formed into a large, flanged or petticoated insulator of irregular cross section would behave quite differently under the same test. For this reason the safest and commonly accepted method of determining the heat and cold shock resistance of a complete commercial insulator is to subject the complete unit to alternating cycles of heat and cold, as from immersion in boiling water to immersion in ice water.

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[3] *Transactions*, Electrochemical Soc., Vol. LXV, pp. 27–34 (1934).

[4] C. W. Parmelee and J. O. Kraehenbuehl, "Mechanical-Electrical Stress Studies of Porcelain Insulator Bodies," University of Illinois *Bulletin, No. 31,* April 2, 1935.

The foregoing is sufficient to indicate that laboratory tests of porcelains are essential, but only for comparing their fulfillment of the general requirements for such material. Any so-called porcelain which such tests show is not insoluble, impervious, highly heat-resistant, oilproof, mechanically strong and of comparatively high dielectric strength does not constitute an acceptable electrical porcelain.

However, slightly varying values obtained from laboratory tests of several acceptable porcelains cannot in fairness be applied to various specific designs in which the dielectric and physical behaviors are mutually dependent. For the performance and quantitative rating of commercial insulators, design tests are requisites even if the insulators are made of a porcelain that ranks high in standard laboratory material tests.

The same conditions apply to the molded materials extensively used in electrical insulation. While standard laboratory tests are valuable and necessary to rate and classify various hot- and cold-molded materials and to determine whether or not a submitted material qualifies for one or another of several classifications, the unit values derived from such tests are not mathematically applicable to all forms and sizes of molded pieces.

In thin-walled molded boxes in which the material must flow a considerable distance in the mold during closure, the physical and dielectric strength of the walls is not as great as that of the bottom of the piece which has been subjected to direct pressure without considerable displacement of material.

In describing improvements in the strength of plastics formed from phenolic resin and fabric for the construction of airscrews, the following statement is made by Aero Research, Limited, of England in a recent article in *The Aeroplane*:[5]

> "Hooke's law is obeyed only up to about 2000 lb. per sq. in. Above this stress the curve departs from linearity at an increasing rate and the material begins to creep under a steady load. At stresses of about 5000 to 6000 lb. per sq. in. the creep becomes so marked that the extensions observed depend very much on the time taken in making the measurements.
>
> "The ultimate tensile strength of such a material is about 14,000 lb. per. sq. in. in the most favorable direction of grain. But the nature of the stress-strain curve shows that this ultimate tensile strength is of little significance in practice because the material is to all intents and purposes already broken soon after the point of departure from Hooke's law."

The article then proceeds to tell of the discovery of a relation between molding pressure and the stress at which departure from Hooke's law occurs. It is quite probable that molding pressure is not equally distributed over all parts of an airscrew during the forming operation.

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[5] "Improving the Creep Stress of Plastics," Aero Research, Ltd., *The Aeroplane*, February, 1936.

Here then is a somewhat involved condition in the field of resin-bonded fabric moldings of such a nature that the conventional laboratory tests for tensile strength and other physical properties applied to standard specimens are not likely to reveal values with sufficient accuracy to justify the elimination of design tests. This type of material is used to a considerable extent in the field of electrical insulation where physical strength is also of importance.

In glass, even after annealing, there are varying inherent strains to a degree dependent upon the design; and neither impact nor heat-shock values will be the same for all designs made from the same glass composition.

Whereas the foregoing illustrations are taken from the field of solid dielectrics, the same principles are involved in other forms of insulation such as those applied as liquids and afterwards converted to film form or even in liquid insulations.

Insulating materials such as varnishes, wire enamels and even pigmented surfacing compounds may have for many of their properties one set of values resulting from the application of standard material tests but somewhat different unit values when tested in place. The reason for this is that films under either tension or compression behave differently under electrical stresses, wide or abrupt temperature changes and exposure to high temperatures. It would be practically impossible to conduct laboratory tests of sufficient scope to forecast conclusively the precise behavior of film-forming materials under all conditions of (*a*) the shapes of wires or coils, (*b*) the degree of stretching, twisting or compression to which such wire or coils may be subjected during manufacturing operations, and (*c*) under the varied strains of service conditions. Between the initial selection of generally suitable materials by standard material tests and the marketing of the finished article there should always be a design test to check the performance of all component parts under the actual and special conditions to be encountered.

In the field of liquid dielectrics, mineral oils under consideration for use in electrical apparatus should be tested for and largely judged by specific gravity, viscosity, pour point, flash point, presence of mineral acids, presence of sulfur, dielectric strength and other properties, but the effect upon the sludging tendencies by other materials can only be determined by a test of the oil in contact with the other materials it is proposed to use. These other materials, which are likely to have an effect upon the oil, will probably vary in number and nature with every substantially different design so that in reality the sludging test becomes a design test rather than a test to determine the properties inherent in the material itself.

NECESSITY FOR BOTH MATERIAL AND DESIGN TESTS

There are, of course, certain properties of insulating materials that are constant under practically all conditions and therefore may be determined just as well from standard laboratory tests as from design tests. These are the properties not affected by collateral influences such as may be imposed by manufacturing, design or service conditions. Among such properties may be mentioned oil-resistance, fusibility, carbonizing tendencies and chemical stability in water.

Purchasers might impose certain limitations upon the values derived from laboratory or standard specimen tests but should not in all cases use the same values as the basis of acceptance or rejection of finished units. Those buying insulation as such are of course justified in imposing material tests for selection, acceptance and control of uniformity, but those buying finished electrical parts containing insulation are not justified in demanding that any and all insulation while in place or after removal from assembled units must possess the same unit values for each individual property as have been obtained from initial tests upon standard specimens.

The following view of standard tests has been expressed by Mr. P. G. Agnew, Secretary of the American Standards Association:[6]

> "Standards should be developed wherever possible in the logical order, the basic ones and those of most general application coming first, and the more specialized and detailed ones coming later and being based upon the fundamental ones."

This statement could well be applied to the logical relation between material tests and design tests.

Material tests, therefore, are essential (*a*) to establish and compare the properties of various insulating materials and (*b*) to establish the acceptability of a given material with respect to general requirements. For reliability in such material tests standard methods are essential, these methods including shape of specimen, method of applying the tests and form of stating the results.

Design tests to make certain of insulation adequacy are often essential and always advisable after a material has been selected upon the basis of its comparative suitability, as judged by standard material tests, and has been subjected to the alterants inherent in the production and use of specific designs.

Both standard material tests such as sponsored by the A.S.T.M. and standard design tests such as are sponsored by the American Institute of Electrical Engineers and the National Electrical Manufacturers Association are necessary and valuable. Both types of tests should be acceptable to the American Standards Association which provides a means for various organizations to work together in developing national industrial standards.

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[6] P. G. Agnew, "National Standardization in America," *Industrial Standardization and Commercial Standards Monthly*, Vol. 4, No. 7, July, 1933.

DISCUSSION

Mr. H. L. Curtis.[1]—It seems to me that Mr. Barringer's paper can be briefly summarized by stating that the final test on any piece of apparatus is a service test. Take a chair, for example: you wish to know whether it holds you up in a comfortable manner while you are sitting in it. You are not primarily concerned with the materials that were used in its construction. You are concerned with its service as a chair. However, if the designer of a chair had to start from the beginning every time he wanted to use a material in making a chair, we would be in a very bad way. We must have some tests of materials to know how to start, and those tests of materials are of the very utmost importance for any application.

In designing apparatus, it is necessary to start with some knowledge of the materials that are to be used if progressively better apparatus is to be made. The Society performs a very important function by giving the basic information on which to start. A designer must know how to use this information in order to apply it in the design of a particular instrument. In designing a new type of instrument, there may be required a knowledge of materials that is not now available. In some cases the designer may not understand all the conditions to which the materials are to be subjected. This only emphasizes the necessity of increasing our knowledge of the properties of materials.

Mr. F. M. Farmer.[2]—When we write a specification for materials, we do not ordinarily anticipate taking over the job of the designing engineer who applies those materials. It is assumed that the application of these materials will be made by engineers, men who are competent properly to apply the information called for by the specifications and will use their own experience and knowledge in making the necessary correction factors, allowances, etc., in the manufacture of a particular article from that material.

A criticism has been made of the Society's methods of testing porcelain. Apparently, however, the criticism should be directed not at the standard but to its being applied quite outside its intended scope. The scope clearly indicates that the standard applies to porcelain as a material and not to articles made of porcelain. The designer, engineer and user must use their own judgment in applying that standard in the manufacture of porcelain articles, the specification tests for which are likely to be quite different from those for the porcelain material.

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[1] Principal Physicist, National Bureau of Standards, Washington, D. C.
[2] Vice-President and Chief Engineer, Electrical Testing Laboratories, New York City.

MR. DEAN HARVEY.[3]—I think we all agree that a performance test is the best test whenever it can be applied but that cannot always be done and we have to depend upon the chemical or physical material tests as a guide in making application of materials. The committees of the Society, in preparing standard test procedures, have a pretty good idea as to how a method should be applied. But it is important that the scope of the test methods be made as clear as possible as an aid in the application of the methods.

It may be advisable sometimes to include explanatory notes indicating that the method has been intended for application along certain lines rather than certain other lines, so as to help the user of the methods as much as possible in making proper application. Frequently, misunderstandings occur because each does not fully understand the other person's point of view.

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[3] Materials Engineer, Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

CONDITIONING OF INSULATING MATERIALS FOR TEST

By Robert Burns[1]

Synopsis

This paper is in partial response to those complainants among the Society's membership who have contended that the technical and administrative branches of industry are in need of a rational discussion of the benefits to be gained from air conditioning for laboratory purposes.

It discusses the problem of air conditioning as applied specifically to the evaluation of raw materials and apparatus for the purpose of determining whether or not they are suitable for an intended use, or to provide the basis of quality maintenance by engineering specification, or both.

It may be described briefly as being constituted of two general divisions. In the first, scientific and economic bases are developed for illustrating the need for conditioning of specimens before test. Many cases are cited where the lack of control of atmospheric influences results in technical confusion and the needless expenditure of monies.

In the second, practical means of controlling the conditioning of test specimens are given. The various ways of producing and maintaining within precise limits those atmospheric influences which are of widest general significance, namely, relative humidity and temperature, are given; and in addition reference is made to those conditions, such as light, which have a particularly significant effect on specific materials.

The so-called vapor pressure system of humidity control is discussed somewhat in detail since it constitutes the best presently known method of obtaining accurate control of humidified atmosphere combined with low cost. Data from reliable source material on sulfuric acid and saturated salt solutions are included for practical assistance to the laboratory technician.

The Need for Conditioning

In general, it may be said that the electrical and mechanical properties of electrical insulating materials vary with the conditions surrounding their use. The amount of variation depends on the material in question and severity of the conditions. The atmospheric conditions to be encountered have from many years experience been fairly well established and can be predicted with a reasonable degree of accuracy. The usual effect on electrical characteristics is a degradation as the humidity is increased. On the other hand, moderately high temperatures, being ordinarily accompanied by the lowering of the relative humidity, usually result in improved electrical characteristics. That such may not always be true however, is illustrated by certain sealing compounds, ceramics, and other materials which show a marked falling off in insulation resistance as the temperature

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

TABLE I.—APPROXIMATE EFFECTS OF ATMOSPHERIC CONDITIONS ON PROPERTIES OF INSULATING MATERIALS.

MATERIAL	PROPERTY AFFECTED	EXTENT OF EFFECT
Phenol fiber and phenol fabric (laminated phenolic)	Insulation resistance	Resistance under normally dry conditions from 100,000 to 1,000,000 megohms between binding posts 1¼ in. apart. After 4 days at 90 per cent relative humidity and 85 F.—about 200 megohms.
	Shrinkage and cold flow	If conditioned for 3 days at 90 per cent relative humidity and 85 F., the thickness will, upon drying for 1 day at 120 F., and with constant pressure, decrease approximately 2 per cent.
Phenol plastic (molded phenolic)	Insulation resistance	Similar to phenol fiber except that insulation resistance after humidifying is somewhat higher.
	Impact strength	After drying at 120 F. for 48 hr., the impact strength is reduced 25 per cent from the "as received" value in damp summer weather.
	Flexural strength	Under conditions similar to those quoted for impact strength, flexural strength values are reduced 10 to 15 per cent.
Sheet cellulose acetate	Dimensional change	Linear dimensions, after conditioning at 50 per cent relative humidity and 70 F., are 3 per cent greater than during dry winter room conditions.
Hard rubber	Insulation resistance	When tested as outlined for phenol fiber, drop is from 1,000,000 megohms to 3000 megohms.
	Impact strength	When tested similarly to phenol plastic, drop in strength is 2 to 5 per cent.
Mica	Power factor	True values not obtainable unless material is thoroughly dried. 2 hr. at 275 F. recommended.
Cork composition	Density Coefficient of friction Compression Dimensional change	All four properties listed are affected seriously by moisture. For reproducible results, drying at 120 F. for 2 days is recommended before tests are made.
Porcelain	Water absorption	Should be dried at approximately 230 F. for 2 hr. before immersion in water. Unless predried, results will vary up to 20 per cent.
Varnished cloth	Insulation resistance	Affected to a degree similar to phenol fiber and phenol fabric.
Cotton and silk yarns	Insulation resistance	Will be 1000 megohms or more under dry winter conditions. After 4 hr. exposure to 85 per cent relative humidity and 100 F. value will fall to few megohms.
	Tensile strength	Values after 3 hr. at 65 per cent relative humidity and 70 F. will be 15 to 20 per cent less than under normally dry atmospheric conditions.
Condenser paper (rag stock)	Insulation resistance	When humidified similarly to phenol fiber except for 48 hr., resistance will drop from several thousand megohms to 100 megohms.
	Thickness	After conditioning for 16 hr. at 65 per cent relative humidity and 75 F., thickness will be 5 per cent greater than at normally dry room conditions.
Kraft paper (wood)	Insulation resistance	When conditioned similarly to phenol fiber, except for 24 hr., values will drop from several thousand megohms to 40 or 50 megohms.
	Dielectric strength	Values are 10 to 15 per cent lower when tested at 90 per cent relative humidity and 85 F. for one day than under normally dry room conditions.
	Tensile strength	30 to 50 per cent change in strength between normally dry and normally moist conditions.

is increased. Also certain other insulating materials for use in electrical apparatus operating at normally high temperatures, may be affected as to dielectric strength. In the case of mechanical properties of insulating materials, these are quite likely to be decreased by drying out. This is especially true of the shock resistance of fibrous materials.

Temperature and humidity are the two conditions ordinarily affecting insulating materials. However, under certain uses, other conditions may enter, such as the presence of sunlight or immersion in water or other liquids.

In Table I is given a list of materials studied by the Bell Telephone Laboratories showing the properties affected by atmospheric conditions and, in certain cases, the amount by which these properties are affected. It will be seen for instance that the insulation resistance of phenolic laminated products may, under certain conditions of humidity, be but a fraction of the value under dry conditions. Also in the case of phenolic molding materials, the resistance to impact under dry conditions may be reduced by 25 per cent or more from that under moist conditions.

One of the primary purposes of conditioning materials for test is to establish engineering requirements by which satisfactory material may be procured. The selection of the specific type of material in the first place is the result of development work which will have shown which material is satisfactory for the use in question. Following this, specification requirements must be set up which will definitely control the quality of the material within the desired limits. In establishing these requirements consideration must be given not only to the conditions under which the tests are to be made but also to the conditions to which the material has been subjected for some time prior to the tests. In the case of impact strength of molded phenolics, already mentioned, it is obvious that if only actual requirements were stated with no mention of conditions, the requirements would necessarily have to allow for a spread of 25 per cent or more to permit testing at all times of the year. Also in the case of direct-current insulation resistance the spread would necessarily be much greater. It would, of course, be possible to apply a correction based on the condition at the time of test or on an approximation of the condition preceding the test. Experience has shown, however, that this is not practicable.

The obvious course is to subject the material to a conditioning period preceding the test. This conditioning is so arranged in character and extent as to provide a common background for the material regardless of its particular history prior to inspection. Thus reproducible results may be obtained, which is one of the essentials of inspection tests. The conditioning characteristics as to severity and time are not ordinarily chosen to provide complete equilibrium of the material. To do so would, in many cases, be uneconomical in view of the long time required. All that is necessary is to so approach the condition of stability that the variations in results are small as compared with the requirement value. Also the conditioning exposures are usually chosen to approximate, if practicable, the conditions likely to be encountered in actual use. The usual practice, however, is to accelerate the conditioning by providing atmospheres somewhat more severe than those expected in service. This shortens the time of test and provides a factor of safety. Referring again to Table I, there will be noted the conditioning methods that have been applied to each of the materials discussed herein.

In selecting the extent and type of the conditioning exposure it is, of course, necessary to select conditions which will not significantly affect the inherent characteristics of the materials under consideration. It is also desirable that conditions be selected which can be easily and economically controlled.

It is recognized that conditioning involves an added item of expense to inspection procedure, not only in the first cost of conditioning facilities, but in added time in the performance of tests. Nevertheless, conditioning in many cases is essential, for without it the establishment of requirements which will provide any appreciable measure of quality control is impossible.

PRACTICAL CONDITIONING METHODS

Small-Scale Conditioning:

In subjecting materials to adverse atmospheric conditions, good engineering and good economics dictate that the purpose of the test be clearly understood. Since conditioning at best is an expensive procedure, the testing engineer will do well to recognize the economic distinction which many times exists between the answers to the two questions:

1. Is the material good enough for the intended purposes?
2. What is the absolute value?

In determining the tensile strength of paper, for example, which is quite sensitive to atmospheric conditions, maintaining an atmosphere of 65 to 75 per cent relative humidity will probably be satisfactory for acceptance purposes. However, for comparison between two or more laboratories, much closer control would be necessary. In measuring laminated bakelite sheet for insulation resistance, a relative humidity of 88 to 92 per cent at 85 to 90 F. will suffice for acceptance, but for referee purposes not only will the humidity and temperature need close control but a pre-conditioning will be necessary so that all specimens will have a common starting point, cancelling out possible effects of different previous histories.

Having decided just what is to be accomplished the engineer can turn his attention to equipment. Perhaps the smallest practical conditioning chamber is the ordinary desiccator jar. Desiccator jars are portable and have ground covers which eliminate leaks and consequent change in density of acid or salt solutions. They can be placed in constant-temperature rooms or refrigerators. If electrical measurements are to be made, a desiccator jar can be secured fitted with large rubber plugs for lead-out wires connecting to specimens. For insulation the inside of the rubber plug should be coated with a high-melting petroleum wax or ozokerite to confine the leakage to the specimen under test, and care should be taken that the acid or salt solution does not splash. (Sulfuric acid solutions are preferred for insulation work.)

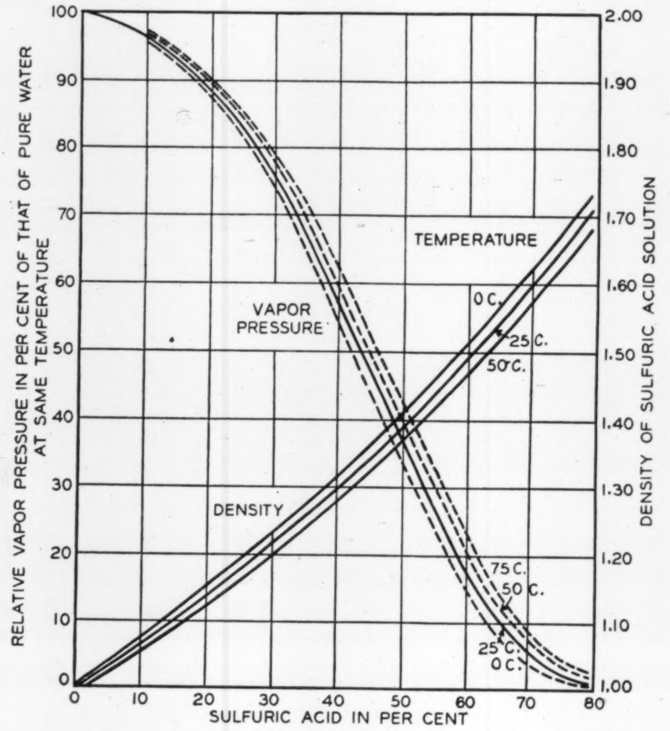

Fig. 1.—Relative Vapor-Pressure Curves for Sulfuric Acid.

However, desiccator jars are not without their disadvantages. The thermal conductivity of glass vessels is of such magnitude as to render the avoidance of condensation almost impossible. This is particularly true at temperatures just above freezing where the dew point or wet bulb depression (at 90 per cent relative humidity) is approximately one degree from the dry bulb temperature, and also at high temperatures, for example 120 F., where the dew point (at 90 per cent relative humidity) is several degrees removed. In addition, it is difficult to measure the actual humidity in a desiccator jar. As this can be accomplished only by rather elaborate apparatus the usual procedure consists of calculating relative humidity

TABLE II.—PERCENTAGE RELATIVE HUMIDITIES OVER SATURATED SALT SOLUTIONS—AND RELATED INFORMATION.

Solid Phase		10 C. (50 F.)	20 C. (68 F.)	30 C. (86 F.)	40 C. (104 F.)	100 C. (212 F.)	Price per Pound	Remarks on Suitability for Humidity Control
Lead Nitrate $Pb(NO_3)_2$	a[a].	99	98	96.5	95.5	88.4[h]	$0.42	Solution slightly acidic due to hydrolysis—probably stable
	b[a].	30.8	34.3	37.8	41.0	158[h]		
Potassium Sulfate K_2SO_4	a..	98.2	97.1	96.6	96.1	93[i]	0.24	Excellent
	b..	8.44	10.0	11.5	13.17	24[i]		
Disodium Phosphate $Na_2HPO_4 \cdot 12H_2O$	a..	.....	95	.....	.....	.....	0.37	Good only below 29.6 C.
	b..	.....	8.51	.....	.....	.....		Transition point
Potassium Nitrate KNO_3	a..	95.1	94.2	92.5	89.4	61.2[j]	0.50	Solution has large temperature coefficient, probably stable
	b..	17.7	24	31.4	39.1	75[j]		
Di-potassium Phosphate—K_2HPO_4	a..	.....	92	.....	.....	.....	1.25	Satisfactory
Sodium Carbonate $Na_2CO_3 \cdot 10\ H_2O$	a..	99	92	87	.....	.....	0.37	Good only below 32 C.
	b..	11.2	21.5	29	.....	.....		Transition point
Sodium Tartrate $Na_2C_4H_4O_6 \cdot H_2O$	a..	.....	91.2	91.1	90.7	.....	0.96	Good only below 40 C.
	b..	.....	....	.....	.....	.....		Transition point
Zinc Sulfate $ZnSO_4 \cdot 7H_2O$	a..	94.7[b]	90	88.5[f]	84	.....	0.40	Excellent below transition point—about 40 C.
	b..	34[b]	35	36.7[f]	41	.....		
Barium Chloride $BaCl_2 \cdot 2H_2O$	a..	.....	....	88[g]	.....	.....	0.37	Small temperature coefficient
	b..	25	26.3	26.5[g]	29	.....		Somewhat poisonous
Potassium Chromate K_2CrO_4	a..	.....	88	.....	.....	.....	0.73	Excellent—small temperature coefficient
	b..	38	38.6	39.5	40	.....		
Sodium Potassium Tartrate (Rochelle salts) $NaKC_4H_4O_6 \cdot 4H_2O$	a..	87.5	87.2	87.1	86.0	.....	0.68	Good only to 40 C.
	b..	30	37	44	51	.....		
Potassium Chloride KCl	a..	87.4	86.3	84.5	82.8	74.7	0.37	Excellent
	b..	23.8	25.6	27.2	28.7	35.9		
Potassium Hydrogen Sulfate $KHSO_4$	a..	.....	86	.....	.....	.....	0.50	Solution is acidic, but stable
Potassium Bromide KB_r	a..	86	84	82	80	69.2	0.80	Satisfactory
	b..	38.3	39.4	42	43.2	51.2		
Sodium Carbonate $Na_2CO_3 \cdot H_2O$	a..	.....	....	.....	78.1	82.2	0.40	Good only *above* 32 C.
	b..	.....	....	.....	33.2	31		
Sodium Chloride $NaCl$	a..	76.9	75.8	75.1	74.4	73.7	0.30	Excellent—practically constant humidity
	b..	26.3	26.38	26.5	26.65	28.1		
Potassium Tartrate $K_2C_4H_4O_6 \cdot \frac{1}{2}\ H_2O$	a..	.....	74.1	72.9	72.1	.....	1.15	Very good
	b..	.....	....	.....	.....	.....		
Mixture—(NH_4Cl and KNO_3)	a..	.....	72.6	68.6	.....	.....	....	Probably does not evolve any ammonia fumes
Cobalt Chloride $CoCl_2 \cdot 6H_2O$	a..	72.5[c]	67.3	61.9	56.6	49[k]	2.00	Good up to 55 C.—Transition point
	b..	49.7[c]	52	55	58	61.7[k]		Large temperature coefficient
Sodium Bromide $NaBr \cdot 2H_2O$	a..	.....	58	.....	.....	.....	0.80	Good to 55 C. Solubility shows practically no change above 55 C.
Sodium Dichromate $Na_2Cr_2O_7 \cdot 2H_2O$	a..	.....	52	.....	.....	.....	0.24	Excellent—low temperature coefficient
	b..	63	64.3	66.3	68.8	.....		
Calcium Nitrate $Ca(NO_3)_2 \cdot 4H_2O$	a..	66[d]	56	51	46	.....	0.70	Good up to 42.4 C.
	b..	48.2[d]	54.8	59.5	67	.....		
Potassium Carbonate $K_2CO_3 \cdot 2\ H_2O$	a..	47[d]	44[e]	43[g]	40	.....	0.50	Excellent
	b..	51.2[d]	52.5[e]	53.2[g]	53.9	.....		
Mixture—$NaCl$ and $KClO_3$	a..	.....	....	.....	.....	36.6[l]	....	Excellent
Magnesium Chloride $MgCl_2 \cdot 2H_2O$	a..	34.7	33.1	31.7	31.3	.....	0.40	Satisfactory
	b..	34.9	35.3	36	36.5	.....		
Mixture—$NaCl$ and KNO_3		32.6 per cent at 16.4 C.					....	Satisfactory
Mixture—$NaCl, KNO_3$ and $NaNO_3$		30.5 per cent at 16.4 C.					....	Satisfactory
Lithium Chloride $LiCl \cdot H_2O$		15 per cent at 20 C.					3.20	Good above 12.5 C.
$H_3PO_4 \cdot \frac{1}{2}H_2O$		9 per cent at 20 C.					0.96	Extremely corrosive

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[a] a = percentage relative humidity; b = solubility, grams of anhydrous salt per 100 g. of solution.
[b] 5 C. (40 F.) [d] 0 C. (32 F.) [f] 25 C. (77 F.) [h] 103.5 C. (218 F.) [j] 110 C. (230 F.) [l] 16.4 C. (61.5 F.)
[c] 18 C. (65 F.) [e] 18.5 C. (65 F.) [g] 24.5 C. (76 F.) [i] 102.1 C. (215 F.) [k] 55 C. (131 F.)

from the acid or salt solution used. See Fig. 1 and Tables II and III, described in detail later.

Another point of importance is that the vapor pressure in a desiccator jar where the cover is sealed with petrolatum in the usual manner actually builds up. This pressure, for a relative humidity of 90 per cent and temperature of 85 F. is about 1 in. of mercury above atmosphere, and is sufficient at 120 F. to require that the cover be fastened in position. This, of course, is an artificial condition and humidity reactions obtained with desiccator jars frequently conflict with those produced by the ordinary humidity tank which is at atmospheric pressure due to leakage. If the desiccator cover is not sealed, then the contained relative humidity becomes largely guesswork.

TABLE III.—SELECTED LIST OF VAPOR PRESSURES.

SALT	RELATIVE HUMIDITY OF SATURATED SALT SOLUTIONS AT 20 C., PER CENT	SALT	RELATIVE HUMIDITY OF SATURATED SALT SOLUTIONS AT 20 C., PER CENT
$Pb(NO_3)_2$	98.	$K_2C_4H_4O_6 \cdot \frac{1}{2}H_2O$	74.1
K_2SO_4	97.1	$CoCl_2 \cdot 6H_2O$	67.3
$Na_2HPO_4 \cdot 12H_2O$	95.	$NaBr \cdot 2H_2O$	58.
KNO_3	94.2	$Na_2Cr_2O_7 \cdot 2H_2O$	52.
K_2HPO_4	92.	$Ca(NO_3)_2 \cdot 4H_2O$	51. (24.5 C.)
$Na_2CO_3 \cdot 10H_2O$	92. (18.5 C.)	$K_2CO_3 \cdot 2H_2O$	43. (24.5 C.)
$Na_2C_4H_4O_6 \cdot 2H_2O$	91.2	$Zn(NO_3)_2 \cdot 6H_2O$	42.
$ZnSO_4 \cdot 7H_2O$	90.	$NaCl$ and $KClO_3$	36.58 (16.39 C.)
$BaCl_2 \cdot 2H_2O$	88.	CrO_3	35.
K_2CrO_4	88.	$MgCl_2 6H_2O$	33.1
$NaKC_4H_4O_6 \cdot 4H_2O$	87.2	$NaCl$ and KNO_3	32.57 (16.39 C.)
KCl	86.3	$NaCl$, KNO_3 and $NaNO_3$	30.49 (16.39 C.)
$KHSO_4$	86.	$LiCl \cdot H_2O$	15.
KBr	84.	$H_3PO_4 \cdot \frac{1}{2}H_2O$	9.
$NaCl$	75.8		

Naturally, the number of specimens which can be tested at one time in a desiccator jar is extremely limited, and objection is frequently raised that conclusions based on such limited data are not reliable. This is a valid objection in many cases, and where more data are required a larger chamber is necessary.

This can be a well-insulated wooden box lined with tinned copper and equipped with a tightly fitting door. A conveniently sized box would be 3 ft. wide, 2 ft. deep, and $1\frac{1}{2}$ ft. high, inside dimensions. A lead tray to contain the humidifying solution should cover most of the bottom and should be 4 in. deep. This will provide adequate volume of solution to permit several tests without changing appreciably the density of the acid solution, or permitting drying up of a salt solution. Pipes may be run from the solution tray to the outside to allow changing of solution without opening the chamber. A small continuously operated fan with motor located outside the box will provide general circulation for the chamber and in particular, movement of air for the wet bulb. The necessary speed of air past a wet bulb has been established by the U. S. Weather Bureau as 15 ft. per sec. but experience has shown that if the wicking and water are renewed every four or five days, this can be safely reduced to 3 or 4 ft. per sec., especially at high humidities where differences are small and wick evaporation correspondingly slow. This is mentioned because violent circulation of air within a humid chamber is an unnatural condition and militates against obtaining reproducible results.

In a reasonably well-constructed chamber, having obtained a state of equilibrium between the liquid and the space above it, subsequent changes will be due to temperature variations which require the humidifying solutions to take on or give up moisture. It is obvious therefore, that little compromise can be allowed in temperature control. For all practical purposes the temperature of test should not vary more than 2 deg. Fahr. from the nominal, and the chamber should be sufficiently insulated to prevent rapid changes of any amount.

If the heating units are contained in the humidified chamber, care should be taken to avoid hot spots and the specimens should be shielded from direct radiation.

Into one end of the chamber can be built a hard-rubber binding-post panel surfaced on both sides with a high melting point petroleum wax or ozokerite for electrical tests. Wires from specimen electrodes to panel should be bare to provide a dead short circuit if the wires touch rather than a high-resistance leak with insulated wires which might give erroneous results.

The space available in this chamber permits the use of wet and dry bulb thermometers which have been found to constitute the best method of measuring relative humidity. They may be observed through a double glass window in the door. It is recommended that U. S. Weather Bureau publications on measurement of relative humidity be given careful study.

Neither the desiccator jar nor the small wooden chamber can be equipped conveniently for testing below room temperatures. If low temperatures are necessary, such devices should be placed in refrigerated rooms. This should be done wherever possible although practice has shown that an immense amount of valuable information can be obtained without this refinement.

A further development in equipment consists of a humidity chamber such as the one described above entirely enclosed in a temperature-controlled outer box, both chambers being well insulated to prevent in the latter case, influence of changes in room temperature, and in the former, to insure that variations in the outer chamber have a minimum effect on the humidified space. Insulated doors are provided for both inner and outer boxes. In such an arrangement heating and cooling is all accomplished in the air space between the two boxes. Consequently there will be no apparatus in the humidified chamber with the specimens except thermometers and the circulating fan. Figure 2 illustrates a chamber constructed in this manner.

Any of the established methods for producing heating and cooling may be used in connection with this equipment, but it is essential that the inner chamber be well insulated so that the unavoidable variations in the temperature-controlled space between the boxes are reduced to a practically constant value for the humidified chamber. One of the disadvantages of this system is the time necessary to reach temperature equilibrium. This may be shortened by having a lamp or similar heating device in the humidity chamber which will be utilized only during the first hour or two of the test.

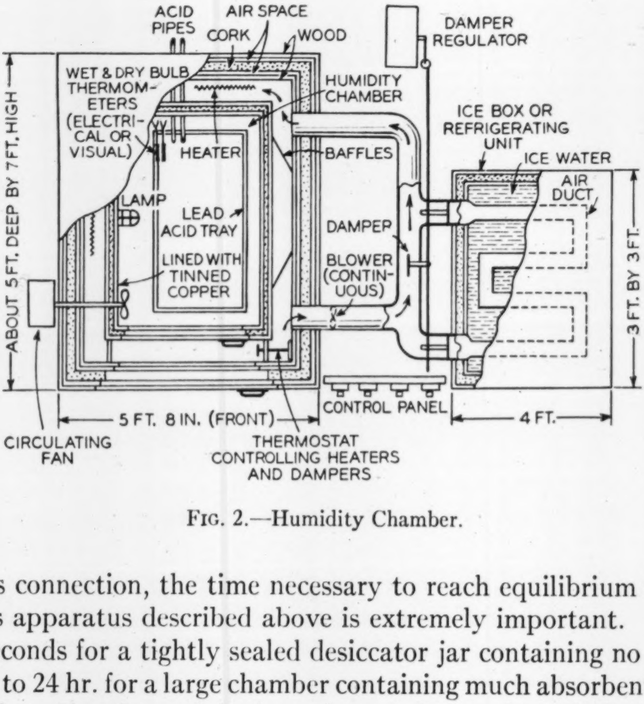
ACID PIPES
AIR SPACE
CORK
WOOD
DAMPER REGULATOR
WET & DRY BULB THERMOMETERS (ELECTRICAL OR VISUAL)
HUMIDITY CHAMBER
ICE BOX OR REFRIGERATING UNIT
ICE WATER
HEATER
BAFFLES
AIR DUCT
LAMP
LEAD ACID TRAY
LINED WITH TINNED COPPER
DAMPER
BLOWER (CONTINUOUS)
ABOUT 5 FT. DEEP BY 7 FT. HIGH
3 FT. BY 3 FT.
5 FT. 8 IN. (FRONT)
CONTROL PANEL
4 FT.
CIRCULATING FAN
THERMOSTAT CONTROLLING HEATERS AND DAMPERS

FIG. 2.—Humidity Chamber.

In this connection, the time necessary to reach equilibrium for any of the various apparatus described above is extremely important. This may be a few seconds for a tightly sealed desiccator jar containing no absorbent specimens, to 24 hr. for a large chamber containing much absorbent material. For example, a chamber employing a large volume of humidifying solution will have the advantage of permitting several tests to be made without an appreciable change in acid-solution density. On the other hand such a volume will lower the rate at which temperature and relative humidity can be raised to the desired point due to the time necessary to heat the solution. It is recommended that each case be given careful consideration before the starting time of test is established. A workable system consists of calling the test "started" when 85 per cent of the desired relative humidity is attained.

Water-Vapor Systems:

The two most widely used methods for maintaining humidity in closed systems of the type described above are those using aqueous solutions of sulfuric acid, and those depending upon saturated salt solutions.

Sulfuric acid solutions have many advantages over other materials which might conceivably be used for this purpose. Homogeneous solutions varying from 0 to 100 per cent water can be obtained; the vapor pressure of these solutions has been more accurately determined than for many other concentrated solutions; the composition, and hence the vapor pressure of the solutions can be quickly and accurately determined by measuring their density, which varies greatly with changes in composition; their relative vapor pressure (per cent of that of pure water at the same temperature) varies but little with wide changes in temperature; they come to equilibrium rapidly with the surrounding atmosphere; the sulfuric acid itself exerts no appreciable vapor pressure, the most delicate chemical tests having failed to detect the slightest indication of the presence of acid in the humid air; and finally, material of adequate purity is cheap and readily obtainable. Figure 1 gives the relative vapor pressure curves for sulfuric acid from which the proper density of acid may be chosen for any desired relative humidity.

Among the disadvantages of acid solutions can be mentioned the unavoidable hazard in handling. This is not great, so far as the solution is concerned, being manifested principally in an occasional acid-burned garment. The acid solution should be prepared, of course, only by those experienced in handling dangerous chemicals.

Continued use of an acid solution results in change of density, due to opening the chamber for replacement of specimens, absorption by specimens, and leakage. Frequent checks of density should therefore be made, especially in small jars or boxes where it is not practicable to measure the humidity by wet- and dry-bulb thermometers.

This brings the discussion to the saturated salt solutions, where the loss of water produces no change in humidity, unless drying actually occurs, which is easily avoided. Most saturated salt solutions exhibit rather large variations in humidity with temperature, a disadvantage not shared by the sulfuric acid solutions. This, however, is not always a valid objection to the use of saturated salt solutions for humidity control since it is necessary for other reasons to control the temperature. Similarly to an acid solution, if the temperature over a saturated salt solution is controlled, the humidity is also controlled. The saturated solution of sodium chloride is a marked exception to the temperature-humidity effect. Between 20 and 100 C. (68 and 212 F.), the variation in relative humidity (75.8 to 73.7 per cent) is comparatively small because of the slight change in solubility of this salt with temperature.

It is also desirable to have other saturated salt solutions covering the range of humidity in which electrical tests are usually made (from 65 to 95 per cent). A survey of the literature indicates that it is possible to provide humidities over practically the whole range from 10 to 100 per cent relative humidity, with saturated solutions of various substances, but few of these exhibit the small change of solubility with temperature shown by sodium chloride. Table II gives humidities calculated from the vapor pressures of saturated solutions of a number of substances.

The table includes the chemical formulas of the solid phase in contact with the saturated solutions of these salts; and, for a series of temperatures from 10 to 40 C., the percentage relative humidity is given in the horizontal columns designated (*a*), and the solubility expressed in grams of anhydrous salt per 100 g. of solution, in the horizontal columns (*b*). An approximate price estimate is included, together with information regarding such details as stability, and danger from poison or handling. Since an important use for these solutions is in humidity control for electrical measurements of hygroscopic materials, all salts which might evolve corrosive fumes were eliminated, but it should be emphasized that the salt solutions are corrosive in contact with many metals, and they should be kept in glass dishes in the humidity apparatus. It should also be kept in mind that under certain conditions the salt crystals will exhibit a tendency to creep. Cases have been reported where this creepage continued over the edge of the vessel and up the side of the chamber. Under these conditions test results are of extremely doubtful value and consequently creepage, to any extent, is to be carefully avoided.

In Table III is a list of salts given to aid in quickly selecting a salt for a given range of humidity.

It must be kept in mind that the data in Tables II and III and in Fig. 1 represent results obtained under theoretical equilibrium conditions and in general indicate relative humidities 2 to 5 per cent higher than can be obtained in a large humidity tank such as that used for usual laboratory investigation. The actual humidity a given saturated salt solution or acid solution will provide in a specific piece of equipment will depend upon the design of the equipment and the amount of leakage. This, of course, can best be determined by direct measurement after equilibrium has been established.

It will be noticed, in discussing control of humidity by acid or salt solutions, the terms relative vapor pressure and relative humidity are used in a corollary sense. The laboratory engineer should keep in mind, however, that in the usual humidity chamber no actual pressure above the surrounding atmosphere is built up.

Large-Scale Conditioning:

The largest known humidity chamber using the vapor-pressure equilibrium method has a cubic capacity of approximately 170 cu. ft. While larger units could no doubt be built, the tendency, where more space is required, is to take the natural next step which would consist of a room large enough to permit the testing engineer to enter. This in turn means that the room must be sufficiently large that normal opening and closing of the door will not seriously affect the conditioned atmosphere. Roughly, the smallest room should be at least 8 by 12 ft. with a 9 ft. ceiling. Such a room, insulated on all six sides will cost from $6,000 up.

Conditioning of a room must be accomplished, of course, by a system more rapid and more powerful than by the relatively slow evaporation of water. Travel in and out of the room, and leakage through the walls make necessary a humidification method which will more than compensate for anything short of leaving the door permanently open.

The construction of such rooms and the manufacture of equipment for their maintenance is such a highly specialized field that it is not usually advisable to attempt their construction with engineering personnel not trained for this work. Several concerns make reliable equipment for the purpose and their services are recommended.

Briefly, the room conditioning system consists of drawing air from the outside and chilling it to saturation at such a temperature that when subsequently heated to the desired final temperature it will have the desired humidity. This is not so complicated as it seems although the apparatus for reducing it to practice is far from simple. For example, suppose the outside conditions are 50 per cent relative humidity at 50 F. and it is desired to condition the room with 90 per cent relative humidity at 80 F. In order to obtain the latter state we must have in the space within the room about 20 g. of moisture per kilogram of dry air. Therefore, the incoming air will be passed through a water spray heated to a temperature of approximately 77 F. Then, by subsequently raising the temperature of the saturated air to 80 F. the desired humidity is obtained. If the outside humidity is 80 per cent at a temperature of 70 F. and it was desired to maintain 65 per cent at 70 F. in the room, the air would be saturated at 57 F. and then heated to 70 F. obtaining the required conditions. If a still lower humidity is needed, say 40 per cent, the water spray would be chilled to 44 F., then by warming to 70 F. humidity of 40 per cent would result.

This system is generally known as dew-point control and is the most practical method of controlling rooms for materials testing. A humidity room with this system can be controlled to ±2 per cent relative humidity.

Drying:

Another system of preparing specimens of materials for test is drying by heating. This is the common method of obtaining very low humidities and is usually accomplished by ovens. Much remains to be done in this field. Ovens now commercially available vary from those with practically no circulation to those with a vigorous blast of air, and since it has been definitely established that the amount of air circulation has an appreciable effect on test results, it is hoped that the future will witness some standardization of this apparatus.

For normal drying to a condition approximating that of a steam-heated room in winter, two days' exposure to 120 F. will suffice for most insulating materials. For obtaining bone-dry conditions, a few hours at 250 F. will be necessary. In all drying by heat an important requirement is that the temperature will produce no chemical changes in the material which will invalidate the results.

Light:

No discussion of conditioning would be complete without some reference to artificial light sources. Some materials, such as cellulose derivatives, rubber, urea formaldehyde and phenol formaldehyde products, to quote but a few examples, are seriously affected by light. The present knowledge of this subject is quite inadequate but some limited information has been gathered by experience based on laboratory and field experiments.

Artificial light sources may be placed in two general classifications. These are (1) the short wave-length lamps such as the fused quartz mercury arc and (2) the so-called sun lamps, either tungsten-mercury or carbon arcs.

The quartz mercury arc admittedly bears little resemblance, either spectrographically or in intensity, to the solar spectrum. The emissions are intensely spotty and much below the shortest wave lengths found in sunlight. In spite of these disadvantages this lamp is of substantial value in the conditioning of materials for test. Relating its effects to those of sunlight is extremely difficult, of course, but studies over a period of several years with this object in view have made practical approximations possible. A 6-hr. exposure to the emissions of the fused quartz mercury arc at a distance of 18 in. and at a maximum temperature of 110 F. is roughly equivalent to 1 yr. of direct outdoor exposure or 2 to 3 yr. of diffused indoor light.

The so-called sun lamps cover about the same range as the solar spectrum but here again the lines are spotty and direct relation is not possible. The General Electric S-1 lamp, in its effect upon insulating materials, is roughly twice as powerful as strong June sunlight. An exposure of 7 hr. to this lamp is approximately equivalent to one week of outdoor exposure in clear summer weather or to one to two weeks indoors near a window.

The above approximations are based on reactions from exposure to sunlight and the artificial light sources and on quantitative studies of them with a photoelectric cell sensitive to the region of 3100 Å. Sunlight measurements were made during the entire year 1930 at 463 West St., New York City, on a fourth-floor roof approximately 200 ft. from the east bank of the Hudson River, and at Long Beach, L. I. at a point approximately 200 ft. from low water mark.

Conditioning Cycles:

Humidity, drying, and exposure to light may be utilized jointly or severally as the particular demands of the materials under test dictate. However, experience has shown that in many cases judicious combinations will produce results which can be related more closely to actual service conditions and consequently more completely justified on rational grounds. We make frequent use of a cycle which is based on a careful study of indoor humidity conditions throughout the country over a period of several years. This cycle consists of 2 days at 65 per cent relative humidity, 1 day at 75 per cent, 1 day at 85 per cent, 1 day at 90 per cent, 1 day at 85 per cent, 1 day at 75 per cent; all at 85 F. This represents, roughly, a year of indoor conditions and may be repeated for as long as necessary.

A less involved but quite valuable approximation of 1-yr. exposure to indoor conditions consists of a 2-day exposure to 90 per cent relative humidity at 85 F. followed by a 1-day exposure to room conditions followed by a 1-day exposure to 120 F. Two days at 90 per cent relative humidity will produce moisture conditions in the samples roughly equivalent to maximum cumulative summer indoor conditions near an open window. The drying at 120 F. will produce a state of dryness approaching that reached in a heated room in winter.

A complete conditioning cycle combining moisture, drying and light, and found to produce aging similar to that experienced in indoor service conditions consists of (*a*) 2-day exposure to 90 per cent relative humidity at 85 F. (30 C.) (*b*) 1-day exposure to ordinary room conditions (*c*) 16-hr. exposure to a General Electric No. 1 Sun Lamp at 110 F. (45 C.) at distance of 24 to 30 in., and (*d*) 1-day exposure to ordinary room conditions. Each cycle represents 1 yr. of service conditions.

DISCUSSION

Mr. A. C. Walker.[1]—Mr. Burns made one statement which seems to deserve even more emphasis. Unless the conditions of test are controlled adequately, much experimental work may be wasted. Many experimenters have the attitude that approximately constant humidity and temperature are satisfactory. It has been my experience that if these conditions are controlled to a somewhat better degree than is absolutely necessary the results obtained are surprisingly more useful than if the additional care was not taken. This has been particularly true in my work on the insulation resistance of textiles where the electrical properties of textiles are critically dependent on atmospheric conditions. Even dielectric strength, which is considered to be less sensitive to humidity effects than some other electrical properties of insulating materials, may be studied more successfully where careful control is maintained of atmospheric conditions during test.

It seems worth while to mention here that there is a cheap and reliable form of temperature control made by a Philadelphia company. It is readily adjustable to any desired temperature, and the mercury thermostat and operating relay comprising this control can be purchased for about $30. It is foolproof and gives commercially satisfactory operation comparable in precision with the best scientific laboratory equipment. It will operate for years with no servicing or replacement.

Most people think that it is difficult to secure high-precision temperature control, so necessary in close humidity control. It is not, provided modern equipment is used. It can be obtained and the cost is not high.

Mr. T. S. Taylor.[2]—Not only is conditioning important to get correct results as far as comparison of materials is concerned, but it is particularly important in helping to tell what is likely to happen to a material in the worst possible conditions to which it is to be subjected. Material which is very good, as Mr. Burns has said, under standard conditions of test, may be absolutely useless under the worst service conditions. So we are thinking not only of conditioning in so far as being able to get reproducible results, but we are also thinking of conditioning in connection with designing. Conditioning should assist in the classification of materials as to their relative order of value in service.

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

[2] Professor of Physics, Thistle Physical Laboratory, Washington & Jefferson College, Washington, Pa.

MR. C. F. HANSON.[3]—There are a number of reasons for wanting air conditioning. In the case at hand it is assumed that air conditioning is wanted for the purpose of controlling acceptance tests. Air conditioning for this purpose is, of course, closely related to specifications for materials. If the tolerances stated in the specifications are closely limited, then it is necessary to control as nearly as possible all variables which may exist in the test methods. With most insulating materials the percentage relative humidity of the air in the laboratory is a large factor in testing. Under these conditions it is therefore desirable to have controlled relative humidity.

Sometimes it is questionable whether the closely limited tolerances in specifications are warranted. Consider, for example, the dielectric strength of varnished cambric. Some specifications state a dielectric strength of 1200 v. per mil for varnished cambric having a thickness of 0.012 in. In order to test varnished cambric to comply with this requirement, it would be very desirable to conduct a test under controlled relative humidity. because at high relative humidity the test data would in many cases show a dielectric strength less than that specified. This circumstance leads to a controversy with the supplier who may have tested his material with a prevailing lower relative humidity.

There is another aspect to this situation. A lower dielectric strength could be specified without detracting from the usefulness of the varnished cambric. This lower specified value would permit tests to be conducted without a recourse to controlled humidity. It may be questionable whether anything is gained by specifying a high dielectric strength because there are other factors of greater significance to be considered in designing apparatus in which varnished cambric is used as an insulation. The operating stress of varnished cambric in service is only in the order of 40 v. per mil, so it would seem of little advantage to specify an extra 200 v. per mil in a dielectric strength value of 1000 v. per mil.

From the manufacturer's point of view there is a favorable feature in considering controlled humidity. The manufacturer is interested in controlling the quality of his product. He is, therefore, interested in eliminating all variables in the test method so that the test data obtained will be indicative of the uniformity of the manufactured article. However, most manufacturers are aware of these conditions and can make allowance for them.

People at large have become air-conditioning conscious. We have air conditioning for homes, theaters and trains and the advertising literature is emphasizing continuously the advantages of air conditioning. Air conditioning in these places is, of course, for another purpose—human comfort. It may be that, with all this publicity, we as engineers have come to believe that air conditioning for test purposes is indispensable. Perhaps for a lot of materials this is true. For other materials I am not so sure that air conditioning is as essential as the prevailing opinions indicate.

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[3] Electrical Engineer, Irvington Varnish and Insulator Co., Irvington, N. J.

MR. P. H. DIKE.[4] There is another aspect in regard to conditioning; we need to use caution in applying conditioning to specimens, in order to be sure that the conditioning does not produce changes in the specimen that may be permanent. For example, a piece of molded bakelite may be tested for resistivity under conditions of high humidity and then subjected to a dry, hot atmosphere, in order to dry it out thoroughly, say at 120 C. On cooling, the resistivity will be found to be many times greater than before. If it is then replaced in the high humidity conditioning chamber, it will be found that the resistivity has undergone a permanent change—that the electrical qualities of the material have been improved by the baking process.

What I wish to bring out is the fact that we must discriminate between conditioning processes which will give consistent results with repetition on the same specimen, and conditioning processes which are so severe as to change the properties of the material.

MR. L. E. BARRINGER.[5]—I should like to connect some of the points raised in Mr. Burns' paper and the remarks by Mr. Hanson with my paper.[6] One of the properties one could measure equally well in the laboratory or by a design test, either in the field or in the factory, is surface resistivity. Very often the point at which corona appears in insulating materials, perhaps in an outdoor station, cannot be very well determined without control of humidity. The surrounding media have much to do with corona formation. Now, if the practical man decides to put the whole design in the carefully controlled apparatus described by Mr. Burns, we have a third form of test in which are combined laboratory and design tests. In other words, the only way to control humidity is in some such way as Mr. Burns has described and for many purposes that may be extremely important.

Mr. Hanson referred to the dielectric strength of varnished cloth. It is true that most apparatus is over-insulated in places where varnished cloth is used, but very often for mechanical strength in assembly operations more varnished cloth is used than is needed for dielectric strength alone. At the same time, the value of dielectric strength may be necessary at some point. I was reading a patent not long ago in which the inventor claimed a dielectric strength of 1500 v. per mil instead of the usual 1000 v. How can we check that? We do not want to build a complete motor to check it but if it is true perhaps we could use much less varnished cloth and reduce the size of certain machines.

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[4] Research Physicist, Leeds & Northrup Co., Philadelphia, Pa.

[5] Engineer of Insulations, General Electric Co., Schenectady, N. Y.

[6] L. E. Barringer, "Significance of Material Tests and of Design Tests upon Electrical Insulating Materials," see p. 592.

So from whatever point of view you take, you get back to the fact that at some time the *general properties* of materials must be determined, preferably in the laboratory by the use of standardized methods applied to standard test specimens.

MR. F. M. FARMER.[7]—I think we will recognize in this discussion on humidity in connection with our testing work, the experience we have had in most of our A.S.T.M. activities, namely, that our tests are constantly becoming more refined. We gradually increase the precision of the existing tests and add new ones on additional characteristics as we acquire more experience—all in response to the demand of the application engineers.

The engineer's job, you know, is primarily to so design equipment that the most is obtained for the dollar and his constant effort is to use his materials more efficiently and more effectively. It was not so very long ago that we paid little attention to either temperature or humidity in our tests of insulating materials. Recently, however, we are getting more particular about these things. Why? In the first place, we need to make our test conditions more precise in connection with the specification itself in order to reduce the opportunity for controversy where there is some question about whether or not the material complies with the specification. A specification that is all that it should be prescribes all the conditions that will affect the test results so that there can be no question as to the results if the tests are properly made. It is, therefore, necessary in the commercial application of specifications to have the conditions surrounding the tests as precise as is reasonably practicable.

A second and more important reason for giving this matter of conditions such careful attention in connection with insulating materials is the fact that, as Mr. Taylor has pointed out, they are subjected to such a wide range of conditions in service. Heretofore the application engineer has simply had to allow for that wide range of conditions in his design. In other words, the factor of safety had to be big enough to cover the whole range of conditions. In the interests of efficiency and more economic design he wants to cut down that factor as much as he can, but he cannot do so unless he actually knows the effect of the maximum range of conditions to which the material is likely to be subjected.

With respect to this humidity question, there is another reason why its control must be given more attention in the testing of insulating materials. I refer to the electric shock hazard in electrical equipment. With the greatly increased use of electrical devices by the general public, it is becoming more necessary to assure their freedom not only from shocks that might result in personal injury, but even from minor shocks which cause no physical harm but do produce an unfavorable psychological reaction. As a result, a limit for such devices of 0.2 ma. leakage current at 120 v. after 24 hr. exposure to an atmosphere of 85 F. (±5 deg. Fahr.) and not less than 85 per cent relative humidity, is a commonly accepted standard. While the design and construction of the device influences this leakage current, it is obvious that the resistivity of the insulating material is the important factor. So we must expect to have to be able to not only control, within reasonably close limits, the established standard humidity, but also to vary the controlled humidity conditions over a relatively wide range because the application engineer will not only be wanting to know how the material behaved under the standard condition but also how the behavior is affected by departures from the standard condition.

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[7] Vice-President and Chief Engineer, Electrical Testing Laboratories, New York City.

Of course this humidity factor is only one of several but we have to take them one at a time, and as the art progresses and we get more precise in dealing with these things, we will be bringing other factors into the picture in the testing of insulating materials.

MR. ROBERT BURNS.[8]—One of the questions raised by Mr. Hanson is really a question as to whether we are over-engineering our product. I am sure that in most industries we do over-engineer somewhat and until we know more about the materials used, we will have to continue to do so. But if he wants to use materials with any degree of precision, he will have to condition them for the dielectric strength tests. For instance, if he feels that any users want 300 v. per mil, unless he is prepared to meet it by a very wide margin the user will have to tell him under what conditions the 300 v. are meant. Otherwise there will be constant argument. The receiving department or the testing laboratory sends it back to the supplier. The supplier sends it back and claims he has tested it and found it to be over 300 v. per mil. It may easily be due to different conditions. One cannot get a precise measure of value without conditioning.

On the other hand, for a lot of testing work conditioning is not necessary and it would be silly and foolish to do it, and would entail needless expense. There is no doubt but what a lot of materials can be accepted for electrical use without conditioning.

Mr. Dike mentioned permanent changes. In the paper we have warned against any conditioning procedure which might cause permanent chemical changes of material. In regard to Mr. Barringer's remarks on design and material tests, in the A. S. T. M. we are confined to testing materials, but practically everything in the paper is equally applicable to testing apparatus. The use of special conditions for testing apparatus preceded the use of those conditions for testing materials. That is, we used the old Turkish bath type of humidity tank on completed apparatus long before we used it on materials, so that no one need hesitate to put completed apparatus in a humidity tank. That is just as valid a test, and as far as tradition is concerned, more valid than testing of materials.

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[8] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

I think the time will come when the method of conditioning described in this paper will be considered a mild method compared to the old sledgehammer method we used for many years—throwing things in pails of water, and having them in so-called humidity tanks with water splashing all over them. In some cases we even threw them into salt water! The tendency will be toward conditioning methods which more nearly simulate conditions found in the field.

SYMPOSIUM ON THE LIMITATIONS OF LABORATORY AND SERVICE TESTS IN EVALUATING RUBBER PRODUCTS

METHODS OF EVALUATING TIRE PERFORMANCE

By R. D. Evans[1]

Introduction

In the whole category of highly developed engineering structures, there is none which is required to operate under conditions of greater relative distortion than the modern pneumatic tire. It is also true that few, if any, such structures are called upon to withstand more types of destructive stress throughout a useful life of more stress cycles than the tire. Faced with these requirements, the pneumatic tire has evolved as a finely balanced and carefully integrated system of structural compromises.

It is the principal purpose of this paper to discuss some of the methods of analyzing and evaluating the principal components of this highly complicated structure which we call a tire, and to examine the possibilities and limitations of interpreting the results into a reliable picture of its ultimate performance. It is important that any such interpretation be closely related to the type of service in which it will operate.

It is a truism in the industry that a pneumatic tire is not merely a tire. It is primarily a heavy transport tire, or it is a passenger tire, or an airplane tire, or a racing tire, or a tractor tire, or some other one of the numerous specialized types. There are, on the other hand, a number of fundamental requirements which to varying degrees apply to all pneumatic tires. Since the scope of this discussion will not permit covering all features of all kinds of tires, we may well limit our attention to these common fundamentals, with occasional reference to special or individual details of outstanding interest.

Functions of a Tire:

A pneumatic tire has been defined as a rolling cushion, but that definition is much too limited to apply to tires for modern vehicles. True, cushioning is just as necessary as it ever was, but of at least equal or perhaps greater importance are: (1) adequate traction under all sorts of conditions; (2) good cornering power, which provides steering control of the vehicle at high speed, and is a unique property of the pneumatic tire; and (3) the retention of these properties over a satisfactorily long period of service.

To fulfil these requirements, some of them intrinsically contradictory to others, the tire must be flexible here and rigid there; it must readily accept distortion in this direction, but be strongly unyielding in that. The means whereby these several requirements can best be met are not yet fully comprehended. As we investigate each portion of the tire, however, we may, with a considerable measure of enlightenment, find ourselves able to relate its qualities to the function it is required to perform.

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Dimensions:

There are four principal dimensions which are of interest in any discussion of tires:

1. The over-all diameter of the mounted and inflated tire.
2. The section width or diameter, measured from sidewall to sidewall, at the point of maximum width.
3. The rim width, measured from flange to flange.
4. The diameter of the rim.

These dimensions establish the range of capacity of a tire, and to a certain extent have an effect on its performance when operated within that range.

Principal Parts of a Tire:

It is logical to view a pneumatic tire as made up of three major parts: beads, carcass, and tread.

A. The beads are those relatively unyielding and stretchless portions which anchor the tire firmly to the rim of the wheel.

B. The carcass is the flexible cushioning structure which accepts and transmits and distributes the thrusts and stresses arising from the mass and motion of the vehicle.

C. The tread is the abrasion-resisting sheath which protects the carcass, and which, by its texture and pattern, also provides the necessary friction with the roadway to permit satisfactory handling of the vehicle.

ANALYSIS

Assume that the tire chosen for analysis is designed for passenger car service, and adapted in size and rating to one of the medium sized cars of 1935 or 1936 vintage. Such a car will have snappy acceleration, a top speed of 75 to 85 m.p.h., powerful brakes, and nicely poised steering which permits curves to be taken at high speed. Much of the time it will be driven 50 m.p.h. or more, and over many different types of road. Our problem is to estimate the probable performance of the tire under these average conditions.

A. *Beads*

1. *Wire.*—The principal stress on the bead wire is the tension due to inflation pressure. It can be readily calculated. The stress due to the centrifugal effect of the rotating tire can also be calculated, and is a uniformly

distributed tension which adds to the inflation tension. At the top speed of the car, this centrifugal tension may increase by 30 or 40 per cent the bead tension already present due to inflation. The localized and varying stresses due to rotation and to side forces on curves are difficult to analyze mathematically, but have been examined experimentally. Hence it is possible to estimate the maximum bead stresses.

Physical and metallurgical tests on samples of the bead wire will satisfactorily indicate its ability to perform its function, and will show within what limits the operating factor of safety lies.

2. *Filler.*—This is the nearly rigid rubber compound with which the bead wire is matrixed, and serves to consolidate the bundle of wires into a concrete unit.

Inspection will show whether this matrix is properly and uniformly distributed. There should be no places in which the insulation of the wires is inadequate, and at which separation may be prematurely initiated. Not only should the insulation be of perfect integrity, but the adhesion between compound and wire should have a high value.

3. *Flipper.*—As a means of stabilizing the bead, of improving the "weld" between rigid bead wire and flexible cotton carcass, and also of tapering off or graduating the degree of flexing as between carcass and bead, the core is surrounded by one or more loops of fabric called flippers. The flipper is a highly essential component of the tire, and the weight and weave characteristics of the flipper fabric, as well as the height to which its edges extend into the carcass, have an important bearing on the satisfactory performance of the tire as a whole.

4. *Tie-in.*—This part of the bead structure comprises the extensions of the plies which partially or completely enwrap the bead core. Various tire designers have widely varying opinion as to what constitutes effective and adequate tie-in arrangement. Perhaps the only item of general agreement is that, in low-pressure tires, at least one ply must wrap entirely around the bead, and at least one other must cover two of the three sides of the bead. The larger the tire and the higher its operating pressure, more of the total number of its plies must fulfill these requirements.

5. *Chafing Strip.*—One, and in some cases two chafing strips of rugged square-woven fabric, accurately placed to sheath the outside and base of the bead, are considered necessary to afford protection against cutting and abrasion by the rim.

We have now inventoried the bead structure. Further, assume that our laboratory has furnished a complete picture of the adhesion values between the several strata of this structure. Are we then in a position to predict the performance of the ensemble?

The tire engineer would hesitate to attempt any such prediction. Bead action obviously involves a nice gradation or "tapering off" in the flexing as between highly flexible carcass and rigid bead. It is seldom possible to discern whether the correspondingly nice gradation in structural rigidity actually exists.

There is no part of a tire in which careful and precise workmanship is more important than in the bead. Although the design may be good, the materials of high quality, and the adhesions up to par, slovenly workmanship may produce a structure so uneven and poorly graduated that premature failure at localized sectors is highly probable.

Bead Tests:

The preceding discussion indicates that rotative tests are needed to bring out the true situation as regards bead quality.

As a general picture, bead failures do not form a large percentage of total tire removals, and are usually confined to certain unusual types of service. Correspondingly, to obtain bead break-down in rotative tests, either in the laboratory or on the road, a somewhat unusual set of conditions is required. To secure bead failure, or even to develop distress before the tire fails elsewhere, it is necessary that the tire be run with a very high deflection, and simultaneously be subjected to frequently recurrent or continuous side thrusts of considerable magnitude. This latter requirement is difficult to provide on the ordinary types of laboratory testing machines.

In road tests, conditions favorable to the development of bead breakdown may be listed as follows:

1. The tire should be heavily overloaded.
2. The test route should have as many curves as possible.
3. Each curve should be negotiated at the highest speed consistent with safe handling of the vehicle.
4. Center of gravity of vehicle should be as high up as possible.

One final point on bead performance should be emphasized: A type of bead construction, which has proved entirely satisfactory in one style or type of tire, or with a certain type and contour of tread, may be found quite inadequate if embodied in a tire of different type or proportions or of different tread contour or massiveness. This is a very clear illustration of the general proposition that a tire represents a carefully balanced system of functional elements, so closely interrelated that apparently inconsequential changes at one place are frequently found to have an unlooked-for effect in other parts of the structure.

B. Carcass

In the discussion of the carcass, it will be necessary to confine attention to three basic considerations. These are:

1. The properties of the cord. These may be listed as type of fiber, construction, and twist details.

2. The "topography" of the cord, the principal items of which are its count and its path, or angle.

3. The type, mechanical properties, and distribution of the rubber compounds which form the matrix for the cords.

1. *Cord Properties.*—As individual cords are pulled from a tire, only tensile strength and final or cable twist can be definitely determined. Stretch, either as ultimate elongation or on the basis of the stress-strain graph, depends not only on the original characteristics of the cord, but also on its history during the several stages of processing.

By careful and thorough degumming, the cord may be brought to a condition wherein its gage, unit weight, and the details of its twist and construction can be determined. Likewise the length, and in some cases the type, of staple can be approximately judged. Further refinement can also be made in studying the residual stretch characteristics.

With all these items before him, and on the assumption of standard workmanship, the experienced tire engineer is able to make a fairly reliable estimate of the probable fatigue resistance and bruise resistance and general adequacy of the cord with which he is concerned.

The preceding statement applies when the tire is destined for ordinary passenger car service. As for tires involved in very severe types of operation, due to heavy overloads or to high speeds, or to any condition in which extreme temperature obtains, these items of analysis are definitely insufficient to predicate performance. There seems to be involved here a more basic and intrinsic quality of the cotton fiber itself, some fundamental temper and toughness which may not be fully portrayed by the ordinary tests, and which is discoverable only by large-scale experience with material which continues true to type year after year and crop after crop.

2. *Topography.*—Cord angle is to be recognized as the most important single factor on which the major rolling characteristics of highway tires depend. Thus, the lateral stability or the cornering power of a tire varies considerably with small changes of cord angle. This is the property which provides suitable steering and handling power to the vehicle driver; its insufficiency makes modern high-speed highway travel unsafe and even impossible. Cord angle, presumably through the mechanism of stability, also has an important bearing on the rate and type of treadwear, to be discussed later.

In the reverse sense, cord angle largely determines the cushioning or enveloping power of tires. Thus it follows that the requirements of cornering and of cushioning are contradictory, and the compromise between these two demands limits the allowable range of cord angle to a very small value. Angle also enters the picture of the cord's resistance to fatigue; the tire designer is very fortunate in that the compromise range, as specified above, is also not far from the optimum angle for good over-all durability. If the cord angle of a tire falls outside this range, unsatisfactory operation in one or more respects can usually be expected.

Since the cord is the principal structural component of a tire, a high count, with cords close-packed, predicates a good degree of ruggedness and resistance to fatigue and bruise.

Conversely, if the count is too high, with an insufficient amount of gum matrix between adjacent cords, certain types of separation are enhanced. A low count is also held to be conducive to a lower operating temperature. In the case of passenger tires, modern methods of building have a tendency to reduce, or "open up" the count, so that we are usually more concerned with having the count too low than too high. Experience is the only reliable guide on this point, but a survey of modern standard-grade tires shows that the space allotted to each cord varies from 120 to 150 per cent of its gage or diameter as measured in the degummed state. In the big majority of cases this range lies between 125 and 135 per cent.

In large tires, or any tire operating at high temperature, separation on top of the outside ply is frequently encountered. Definite improvement in this respect is provided by a considerable lowering of the count in the last, or in the last two plies; but a stiff price is paid by the lowering of the resistance to bruising when the tires are required not only to travel fast and therefore hot, but also to negotiate gravel or uneven and broken-up roads at the same time.

3. *Rubber Compounds.*—A long treatise would fail to cover completely the ramifications of the subject of rubber compounds. Suffice it to say here that chemical analysis and the various mechanical tests, while very useful, are inadequate to give more than a preliminary picture of the total requirements of carcass compounds, which include resistance to oxidation and to overcure under prolonged high temperature.

As to the distribution of the carcass rubber, the effect of cord count has already been outlined. Similar considerations apply to the inter-ply gum. Insufficient inter-ply matrix is a reliable index of premature separation, no matter how high the quality of the compound itself. The greatest need for inter-ply matrix is in the shoulder zone where the flexing reaches its maximum. The region near the bead has not only less need, but also less room, for matrix. Hence, we arrive at the practice of using squeegees, or auxiliary strata of compound, applied either as bands over the whole crown or as ribbons at the shoulder. This is an attempt to distribute the gum to optimum advantage.

Too much matrix, particularly in the mid-tread zone, may be a definite disadvantage, because by unduly thickening the whole casing it reduces its ability to dissipate the hysteresis heat which is the chief problem in high-speed service.

Carcass Tests:

Of the three major divisions of a tire, the carcass is the only one whose over-all performance can be reliably estimated on the basis of inflated-tire tests in the laboratory.

As for rotative tests, various types of testing machines have been developed to give almost any desired type of action on the tire. Pulleys or flywheels are of various diameters and curvatures and are either smooth or fitted with a wide variety of cleats or obstacles. They are capable of operation at any desired speed up to those of a racing car. Hence, we may set up conditions in which almost any known type of carcass failure may be emphasized. One combination may bring out the resistance of the cord to flexing fatigue, and permit the comparison of or evaluation of different tires from this standpoint. Another combination can be provided to cause interply separation; still another will develop the separation on top of the carcass or in the breaker. Likewise, the relative ability of tires to dissipate the respective amounts of heat generated by high-speed running, and thus to remain intact under the respective centrifugal forces, can be satisfactorily judged. With modern tires, this last type of test is of increasing importance.

High-class workmanship in all of its phases is no less important in connection with carcass than with beads. While detailed examination of the dissected tire will uncover such things as variable count, irregular cord angle, and non-uniform gum distribution, rotative tests under closely controlled laboratory conditions are peculiarly effective in bringing out the great importance of these factors. In group testing (and no rotative carcass test of any kind should be given consideration unless at least 3 tires comprise each group), the variation in performance beween various tires of a group, as well as the point-to-point variation in an individual tire, is an important index of the quality of workmanship.

Mention should also be made of certain useful tests which are nonrotative but involve mounted and inflated tires. In addition to the usual load-deflection determinations, methods have been worked out for measuring the relative lateral stability and the cushioning or enveloping power ratings of tires. After a sufficient background of comparison has been built up, these laboratory methods can be definitely relied on to give an undistorted picture of the road behavior of tires in these several respects. A substantial saving in the cost of road tests can thus be realized.

Bruising is a hazard to which practically all types of tires are subject, and is a very important cause of tire removals. We believe that large-scale road tests, over test routes which contain a wide variety of bruise-producing conditions, are the most satisfactory as well as surest way to establish the value of such factors as extra plies, or breakers, or massive treads, to mention only a few of the items which contribute to better bruise resistance.

In the effort to get away from the heavy cost of road tests, a large amount of work has been done in the development of laboratory bruise tests. Certain tests of this kind are now specified laboratory routine. They undoubtedly give a useful indication of the efficacy of certain items of structure and of various features of design.

C. *Tread*

As long as the tire engineer busies himself with beads and carcass, those utilitarian but hidden components, he is satisfied to confine his efforts within the bounds of function and performance. However, as soon as he becomes concerned with the enveloping sheath which completely hides these technicals efforts, his hitherto suppressed aesthetic nature begins to yearn toward a more abundant life. So we have, in treads, a mixture of technics with aesthetics, and our attempts to arrive at a well-balanced estimate of treads may be in danger of becoming entangled in this mixture.

Accepting, for the moment, that tires may be, and by all means should be, sturdy looking, or handsome, or fleet-looking, or rugged in appearance, or dignified, let us select out the prosaic technics, and consider the following two aspects of tread and sidewalls:

1. Compound.
2. Design and contour.

Tread wear remains, as it has been for years, the largest cause of tire removals. Evaluation of the rate of tread wear is one of the principal items on the docket of the tire test engineer.

1. *Compound.*—Resistance to abrasion is the most important, although it is not the only property of the tread or sheath compounds of interest.

Tread compound is available in tires in sufficiently large slabs so that test specimens of almost any desired size and shape can be carved out. This fact permits a large variety of mechanical tests to be made on this material. We might, therefore, expect to be able to determine, by such tests, almost any conceivable property; but, as the situation now stands, no fully satisfactory test or combination of tests for abrasion resistance has been developed. We find that these tests can be relied on only for preliminary and very rough classification.

Abrasion tests on rotative laboratory machines are in little better favor, and we may say that practically all testing of this item of tread performance is done on the road.

In tires which operate at a high relative deflection, or at a comparatively high temperature, various forms of tread cracking are apt to make their appearance. While these cracks are related to the configuration or pattern of the tread, they also very definitely depend on the qualities of the compound as well. Rotative laboratory tests provide an excellent means of studying this particular type of misbehavior, and of rating various compounds in relation to a chosen standard.

The preceding statements are to be understood as applying to the sidewall zones of the sheath as well as the part which rolls against the road. In some types of tires these sidewalls are of different compound from the tread zone, either for appearance reasons, or to secure a more satisfactory compromise of properties. In addition to cracking, the sidewalls are also peculiarly subject to sun-checking or "weathering."

Resistance to weathering is a property which can be rated, either with sections of sidewall or with the tires themselves, by suitable "roof" exposure or by the use of ultra-violet lamps.

2. *Design.*—The reasons for molding a grooved design or pattern into the tread portion of a tire are generally understood and accepted. The variety of such patterns or designs is indeed great, and numerous factors contribute to the selection of a pattern which the tire engineer believes will be the most acceptable on his particular product. We are concerned here, however, not with an inventory of these original considerations, but with how the tread itself is going to wear in service.

From the standpoint of tread design, both the rate and the type of wear are of interest. We believe that there is no satisfactory measure of these two items, or even approximation thereto, by any method short of extensive and carefully organized road tests. Certain attempts have been made to study, by laboratory methods, the underlying principals which govern tread wear. These methods may be grouped under two general heads: (1) analysis of the distribution of unit pressure over the contact area, and (2) study of tread movement by "scratch plate" analysis. These methods have been of considerable value from a research standpoint, but do not eliminate the necessity of thoroughgoing road tests.

Ever-increasing speed and improved handling characteristics of the cars, together with certain fundamental changes in chassis design, have considerably altered the picture of passenger tire wear during the past few years. In particular, front wheel tires are prone to exhibit unusual patterns of wear, and these patterns depend on the particular model of car involved, as well as on the features of the tread design itself. Obviously, only road tests with a variety of cars can furnish a basis for comprehensive judgment of these factors.

As we examine a section of a tire, we are confronted with three other major aspects of the tread: its thickness; its width; and its contour, or degree of roundness or flatness. The following general observations may indicate how these features are related to performance:

The thicker the tread the longer it wears, although the increase of wear is not in proportion to the increase of thickness. A case of diminishing returns.

A thicker tread gives better protection to the carcass against bruises. On the other hand, a thicker tread increases the fatiguing action in the carcass, both at the shoulder and at the beads.

Thickening the tread enhances all tire troubles due directly to heat, because it raises the temperature by restricting the escape of heat. At high speed the additional centrifugal effect of the heavy tread also adds greatly to the tendency to tread separation or "throwing."

Widening and flattening the tread contour both tend to improve the wear, particularly on driving wheels. Under conditions, either of road or car, which tend to give irregular wear on front wheels, these same changes of tread may increase the irregularity of the wear. Since either widening the tread or flattening it causes an increase of thickness at the shoulder, changes of this kind tend to augment fabric fatigue and ply separation in the carcass.

These facts further emphasize the general proposition that a pneumatic tire is a carefully engineered product, and that even slight departures in the design or configuration of any item must be carefully analyzed and tested, not only for the direct advantage desired, but also for possible adverse effects elsewhere in the structure.

RÉSUMÉ

In the foregoing discussion, the attempt has been made to present, in fair and proper perspective, the possibilities of and limitations in rating tire quality. The principal elements of the tire are briefly described and their respective functions and weaknesses are outlined. The types of test considered necessary are indicated, although the scope of the paper does not permit any detailed description of them.

CONCLUSIONS

Because of the complexity of its structure, the close functional interrelation of its parts, and the wide variety of conditions under which it is required to operate, the performance and endurance characteristics of a pneumatic tire cannot be determined by any single test.

Although numerous laboratory tests, both on sections and on mounted and inflated tires, are informative, and therefore useful to the tire designer in developing his product, no type of such test can, in our opinion, give more than a limited indication of ultimate tire quality. Road tests, extensive in scope, carefully organized in relation to the type of service for which the product is destined, and expertly conducted and analyzed, provide the only sure way whereby the complete and undistorted picture of tire quality can be obtained.

THE TESTING OF RUBBER FOOTWEAR

By W. E. Glancy[1]

Synopsis

The testing of rubber footwear is being practiced on a considerable scale by the manufacturers but few buyers are equipped for such testing.

This paper describes the laboratory and service testing of a large manufacturer.

It is concluded that neither method of testing is a substitute, but that they are complementary to each other and that after results of each method are obtained a high degree of confidence may be placed in laboratory tests in predicting actual wear.

Introduction

The rubber footwear division, unlike some other branches of the rubber industry, such as the insulated wire, automotive parts, and mechanical goods divisions, disposes of its products to distributors and consumers who do not need to consider further fabrication, and who for the most part buy without reference to standard specifications. The United States Government, to be sure, does buy large quantities of footwear and specifications have been written to cover its requirements, but in almost no other case is this true. Accordingly, the good name of the manufacturer, and experience in the use of such goods are very important factors in the making of commitments.

In view of this situation, this paper considers testing from the point of view of the manufacturer rather than of the buyer.

Rubber footwear factories antedate tire factories and the earliest rubber footwear was not vulcanized at all. Later on it was vulcanized in hot-air, wooden box heaters which had no or, at best, very poor circulation. With the advent of improved compounding methods, better equipment and engineering, pressure vulcanizing, and advance in every direction, it was only to be expected that the evaluation of these changes through testing would be a prime requisite for a footwear producer.

Hence, testing on an increasing scale was practiced by all companies. There were some fantastic attempts to duplicate actual wear through machines which would simulate walking. Experience with such devices was rather unsatisfactory and inconclusive, and chemists and control men obtained more valuable information by cutting up the article, testing it by hand for correctness of cure, adhesion of parts, etc.

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[1] Manager of Laboratories, Hood Rubber Co., Inc., Watertown, Mass.

There were no satisfactory machines for the determination of tensile strength of very thin rubber such as found on a rubber shoe upper and so machines were developed for this purpose. A photograph of such a machine is shown in Fig. 1.

This machine has been in service for 25 yr. and is still doing good work. It is used for determining tensile strength and elongation, adhesions, and hysteresis tests. One feature which was unique to this machine for many years is the means of correcting for the thickness of the specimen, and consequently it is possible to reproduce stress-strain curves automatically. The chart on which such a stress-strain curve is drawn indicates the tensile strength at break in pounds per square inch.

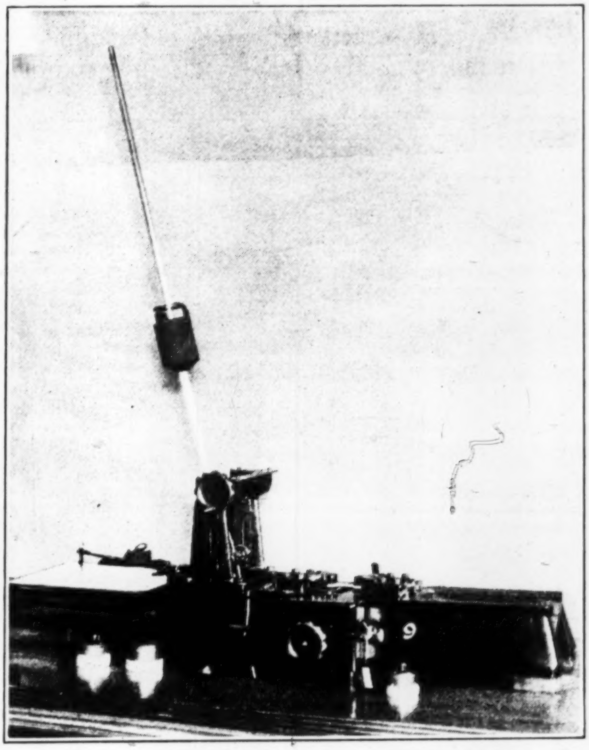

FIG. 1.—View of Hood Rubber Co. Rubber Tester.

The resistance of rubber goods to aging was in the hands of men who used their best judgment in compounding and vulcanizing, but who did not have adequate tools until Dr. Geer developed his artificial aging test. Then it was possible to feel greater assurance about the daily production because results could be obtained in 2 weeks instead of 2 yr.

MODERN LABORATORY TESTING

Today the testing of rubber footwear is as complete a procedure as may be found in other branches of the rubber industry. Both laboratory and service tests are considered to be very necessary helps to the producer. The question has been raised as to which is the more valuable. Experience indicates that neither is a direct substitute for the other, but rather complementary to it. The kind and extent of testing will naturally depend upon the amount of production, the variety of product, and the opinion of management as to a reasonable expenditure for this purpose. New types of goods receive more attention than old established lines. Tests appropriate for one line of goods often do not apply to others.

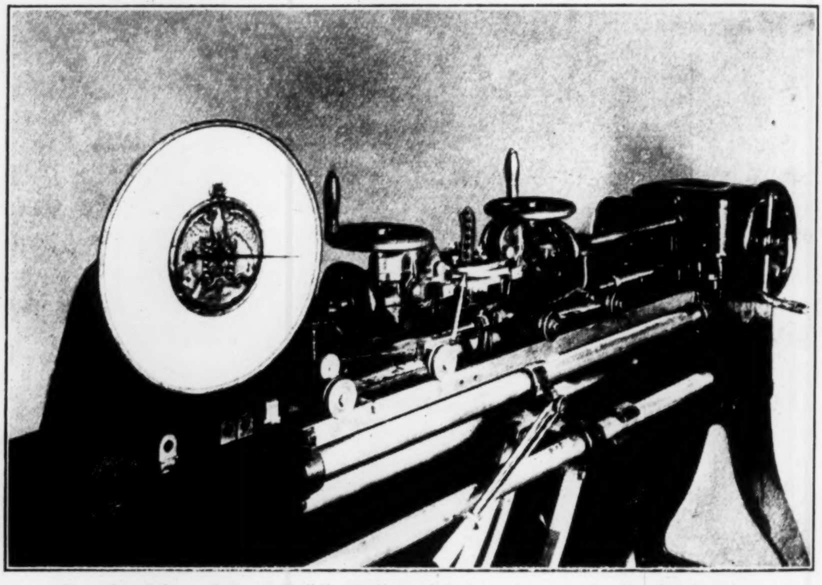

FIG. 2.—View of Scott Fabric Tester with Attachments for Eyelet Testing.

Demand for rubber footwear has caused the production of many kinds of goods. Aside from the common lines of light rubbers, overshoes, tennis shoes, etc., there are special uses, such as for firemen, miners, fishermen, lumbermen, farmers, creamery and garage workers, basketball players, trackmen and sport walkers. Each of these calls for a special type of shoe and the situation is further complicated by the offering of lines for different price classes and for different styles or fashions as in women's footwear.

All types of footwear are examined for correctness of vulcanization. This examination consists of a dissection of the article, the separation of the different parts and the testing of these parts both before and after artificial aging. The rubber parts are tested for tensile strength and elongation at break and adhesion tests are run to prove the bonding of one part to another. The artificial aging usually consists of exposure of the part to an atmosphere of oxygen at 300 lb. per sq. in. at a temperature of 60 or 70 C. for such periods of time as experience dictates (Bierer-Davis method). Parts are also exposed for varying periods of time in a chamber in which hot air circulates in accordance with the Geer method.

Specialists examine the daily product for any visual evidence of factory mishaps.

Boots equipped with safety-toe caps are tested for the force required to press in the reinforced portion. A fabric-testing machine with special attachment is adapted to this use. An alternative method is to drop a heavy iron weight from a height of approximately 4 ft. and measure the depression caused by the weight in striking the toe. A convenient method of measuring this is to insert a cake of paraffin or other wax and note the flattening caused by the impact.

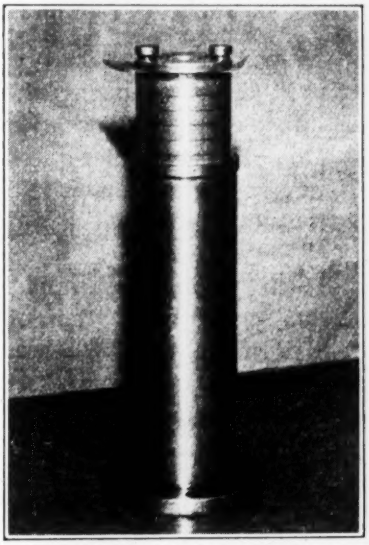

FIG. 3.—View of Gurley Densometer for Canvas Upper "Breathability" Test.

Very flexible fabric-lined shoes are tested for this flexibility feature by measuring the force required to stretch the combined fabric-rubber through a specified distance.

Articles equipped with eyelets are subjected to an eyelet-removing test to note the gripping power of these parts (Fig. 2). This same machine with a slight alteration is used for testing safety-toe caps.

Resistance to cracking through flexing is determined by means of a De Mattia type flexing machine.

Light exposure of dyed fabric uppers, as well as of rubber parts, is considered necessary. Practices differ but natural weathering with and without glass protection, ultraviolet arc light, and Fade-O-Meter are all in use

Still another test to which canvas uppers are subjected is the "comfort" or breathability test, which consists of measuring the rate of flow of air through the fabric by means of a Gurley densometer. This machine can be used to measure the time required for 100 cu. cm. of air under constant pressure to pass through a circular area of about 2.9 cm. diameter (1 sq. in.) A photograph of this machine is shown in Fig. 3.

Heels and soles are tested for hardness by means of a Shore durometer.

And then of course resistance to abrasion is of constant interest for all classes of goods. This is determined by cutting out specimens from the article and using some one of the machines which have been developed for this purpose.

SERVICE TESTING

Despite all precautions which may be taken in designing, manufacturing, and laboratory testing, there still remains the question of wear. The tests which have been described are all very useful in determining and maintaining quality, but they do not answer other questions such as correctness of construction, insufficiency or waste of material at certain points, proper fit, or other unexpected results which have a way of developing in new articles.

So it is that basketball teams may be completely equipped with test shoes because of the severe service which they offer. Shoes are sent to Florida to take advantage of the light and heat effects when northern communities are still having winter. Garages and creameries are very suitable places for comparisons of goods which are to be subjected to the softening effects of oils and fats. Fishermen and miners use rubber boots and "pacs" regularly and are very useful for service testing. At times men and young women are hired to walk daily, testing out new types of goods. Such walkers travel over a prescribed course and register at widely separated points to give evidence of their actual service.

And lastly there is a group of boys and girls which often numbers 75 who wear test shoes. Once each week, those who have such test shoes report to have their shoes inspected by trained workers and by others who are interested in the specific tests. In this manner, any tendencies toward poor service are promptly found.

Finally, there comes the interpretation of results and the correlation between service and laboratory tests. Peculiarities of the walker or unusual service conditions sometimes give puzzling results. Such discrepancies may be discovered and allowed for by conducting a sufficient number of tests or by mismating different shoes and then repeating the tests, so that any one type will receive both left and right foot wear by the same walker. Structural weaknesses or poor workmanship come to light in ways common to most of the shoes under test, and may be corrected after proper diagnosis. Poorly wearing rubber compounds may be interpreted in terms of the physical properties of the stocks as shown by laboratory tests. In general these service tests result as might be expected from a study of the laboratory data.

SUMMARY

Laboratory and service testing in the rubber footwear industry are considered desirable and neither is sufficient in itself. If one were compelled to dispense with either, service testing would be retained.

The laboratory, however, offers advantages for any rubber manufacturer, as, for example, (1) quick results, (2) assistance in assuring uniformity and proper aging, (3) positive evaluation of quality free from the variables of service testing, and (4) money-saving alternatives.

After service testing has been carried on and laboratory standards of the same article established, a high degree of confidence may be placed in the laboratory tests for the control of further production.

SIGNIFICANCE OF LABORATORY TESTS IN EVALUATING AUTOMOTIVE RUBBER PARTS

By J. J. Allen[1]

Synopsis

This paper attempts to show the value of various types of physical tests for evaluating automotive parts such as motor mountings, axle bumpers, shackles, fan belts, hose, etc.

There are three groups of testing methods in use at the present time: (1) laboratory tests on samples of rubber cut from the parts, which include tensile strength, modulus of elasticity, elongation, hardness, compression set, abrasion and resilience; (2) laboratory tests on the parts as a whole where one particular service condition is chosen and accelerated tests are made under laboratory conditions; (3) service tests where the parts are installed in the automobile and subjected to regular road or service tests.

These tests are used for two purposes: the design or choice of the right quality of rubber for the particular part; and control testing to check the uniformity of production.

Reference is made to several representative automotive rubber parts, since the field is too large to cover each item in use today.

In an attempt to show the value of various types of physical tests for rubber for evaluating automotive rubber parts, it was felt that the best approach was from the standpoint of testing methods. With this in mind each type of test and its method of use will be discussed and its application for evaluating rubber parts will be shown.

Classification or Types of Tests

The various types of tests that are being applied to rubber products in both the manufacturers and consumers laboratories can be divided into three groups:

1. Laboratory tests made on test samples cut from the product or specially prepared for the purpose.
2. Laboratory tests on the product as a whole.
3. Field service tests.

The first group includes tests such as tensile strength, modulus of elasticity, elongation, permanent set, stress-strain, hardness, compression set, abrasion, resilience and rebound measurement, and flexing. These tests are usually made both before and after various periods of natural and accelerated aging. The rubber samples for each type of test are either prepared specially from the rubber compound to be used in making a given product, or, in many cases, can be cut from the product and ground to the necessary shape. These are strictly laboratory tests but they are very useful for evaluating the rubber compounds that are to be used for various products, providing information as an aid in the choice of a suitable compound for a particular use and also serving as control tests for determining the uniformity of the product after it is in production.

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[1] Chief Chemist, Mechanical Rubber Goods Division, The Firestone Tire and Rubber Co., Akron, Ohio.

In the second group of tests, one particular service condition is chosen and an attempt is made to duplicate it in testing the product rapidly in the laboratory. For example, it would be extremely difficult to develop and control the uniformity of fan belts if the manufacturer had to depend only on actual service tests where many days or even months would be required to get results. Laboratory testing equipment has therefore been developed in which various service conditions are duplicated, the test usually being accelerated by increasing the speed or the loads on the several pulleys. The same is true of motor mountings which are subjected in service to moderate loads and rapid flexing. Usually some type of laboratory flexing test is set up with greater loads than actual service conditions in order to get some idea of the life of the product in a reasonable length of time. In general, this type of test is applicable to parts that are made up of rubber in conjunction with other materials such as metal or fabric.

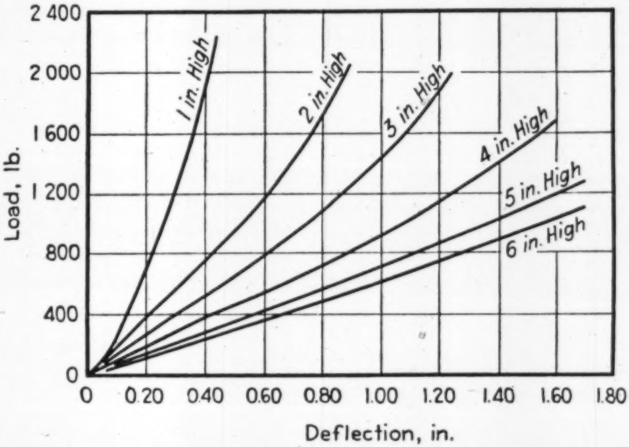

FIG. 1.—Compression Load-Deflection Curves of Rubber Blocks 2 by 2 in. and Height as Shown. A.S.T.M. Hardness No. 70. (Durometer 60.)

The third group, namely, field service tests, involves the testing of the product properly installed in the car. Under these conditions it is subjected to regular road or service tests for thousands of miles of continuous operation. When the car is disassembled the various rubber parts are removed and examined.

RUBBER ENGINE MOUNTINGS

One of the most important uses of rubber in the modern automobile, exclusive of tires, is for engine mountings, practically all of which today are of the rubber-to-metal adhesion type. Rubber mountings are used under conditions of constant compression or shear and vibration to prevent motor vibration from being transferred to the body of the car. A high uniform quality of rubber must be used and maintained. Tests of rubber for engine mountings are, therefore, divided as follows:

1. Tests to determine the proper quality of rubber compound and the proper design of the part.
2. Control tests to determine the uniformity of the product after the part has been put into production.

Under the first type are included tests from all three groups previously mentioned, in particular:

1. Load-deflection tests both on laboratory samples and on actual parts.
2. Hardness tests.
3. Compression-set tests on laboratory samples and on samples cut from the parts.
4. Metal-adhesion tests on laboratory samples and on actual parts.
5. Flexing tests under service conditions in the laboratory on actual parts.

Under the second or control type of tests are those mentioned in group 1 above:

1. Hardness tests on the finished part.
2. Tensile strength and elongation tests either on samples prepared from the rubber compound used or on samples cut from the part.
3. Metal-adhesion tests on the part.
4. Compression-set tests.

Load-Deflection Tests:

Load-deflection curves on samples under compression or shear and hardness measurements on the various types of rubber compounds available for engine mountings are necessary as an aid to the automotive engineer in the proper choice of rubber and in the design of the part. An accurate testing machine for determining the load-deflection curves under compression or shear of rubber blocks of standard dimensions and of actual engine mountings should be a part of every well-equipped laboratory. Load-deflection curves of the rubber compounds, over the entire hardness range, and on blocks varying in size from 1 sq. in. up to 6 sq. in. or even larger should be made. An example of this type of data is shown in Fig. 1. These particular curves were made on blocks of rubber of A.S.T.M. hardness

No. 70, 2 in. square and with heights of 1, 2, 3, 4, 5 and 6 in. Similar curves for stocks of other hardness and blocks of other sizes should also be made. Using this type of data, together with the hardness data of the various compounds, the automotive engineer can determine the approximate volume of rubber and the hardness of the compound desirable for the particular installation. When the proportional weight of the engine to be carried on the mounting is known, together with the amount of torsional movement allowable to dissipate vibration of the motor, reference to load-deflection curves such as these will indicate the volume and hardness of rubber to be used.

The compressive characteristics of a rubber compound can be varied considerably by varying the shape of the part. They can also be varied by bonding the rubber to metal inserts of various shapes. In addition to data on the load-carrying capacity of rubber blocks, it is necessary to determine the load-deflection characteristics either in compression or shear of engine mountings of various designs. Figure 2 shows a cross-section of a standard type of patented biscuit mounting. Figure 3 shows the compression load-deflection curves for this part made with four different hardnesses of compound. The hardness values shown are A.S.T.M. hardness Nos. 50, 70, 100 and 135, corresponding to Shore durometer hardness of 70, 60, 50 and 40 respectively. With this type of mounting it is usually desirable to pre-load the mounting to take up the first $\frac{1}{8}$ in. deflection by pulling the mounting down with bolts. The vibrational action of the rubber occurs, therefore, in the range on the curve between the deflections of 0.125 and

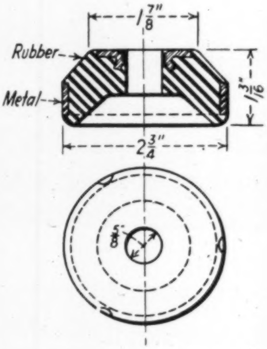

FIG. 2.—Resilient Biscuit-Type Mounting.

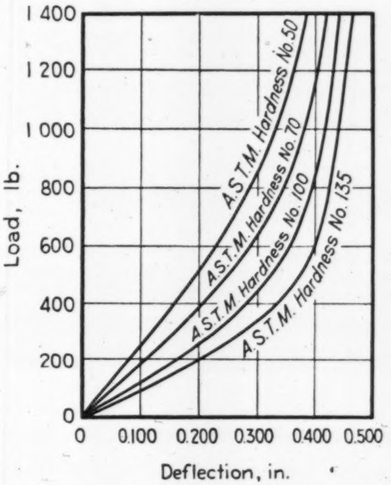

FIG. 3.—Compression Load-Deflection Curves for Biscuit-Type Mountings.

0.25 in. If the vibration characteristics of the engine, static load and the maximum torque load are known, reference to curves such as this will indicate the correct hardness of rubber to be used.

Hardness Tests:

Hardness of rubber has been mentioned. It has become common practice to classify the several compounds available for engine mountings by their hardness. The automotive engineer, having determined the rubber compound he desires, refers to it in terms of its Shore durometer hardness, specifying compound of 30, 40, 50, etc., hard stock. This is unfortunate and leads to many errors due to the variation in readings obtained with different durometers and between different operators.

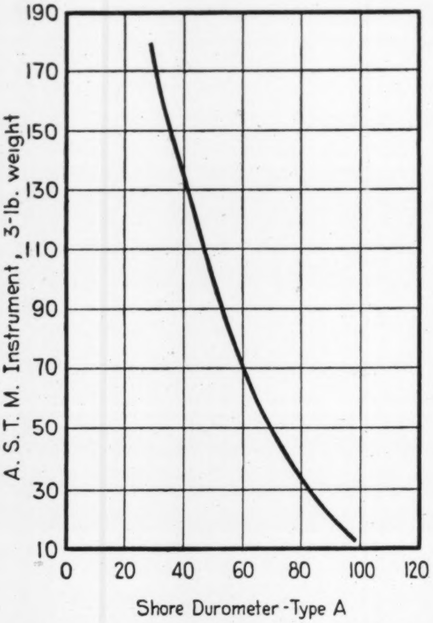

FIG. 4.—Comparison of A.S.T.M. Hardness and Shore Durometer.

Recognizing the importance of laboratory hardness tests, the Society has developed a standard dead-weight instrument and method of using it. This is covered in the A.S.T.M. Standard Method of Test for Hardness of Rubber (D 314 – 34).[2] The hardness of rubber shall be considered as that property by virtue of which the surface and adjoining layers resist indentation when subjected to a definite pressure of 3 lb. ±0.25 oz. by an indentation element, the contact surface of which conforms in shape to a portion of a true spherical surface $\frac{3}{32}$ in. ±0.0005 in. in diameter. The test specimens shall be at least $\frac{1}{2}$ in. in thickness. This instrument is not portable and should be used as a control instrument for checking compound hardnesses in the laboratory and for calibrating the other portable instruments used for product control.

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[2] 1934 Supplement to Book of A.S.T.M. Standards, p. 167.

The curve in Fig. 4 shows the relationship of the A.S.T.M. hardness to that of an average Shore durometer.

The hardness testing of rubber is extremely important and is one of the tests most frequently used by rubber laboratories and inspection departments. The measurement of hardness is a test that can be made on most finished products without harming or destroying the part. It is used, therefore, as a control test to determine whether the correct compound has been used and whether the correct degree of vulcanization has been obtained. Many controversies between manufacturer and consumer arise with reference to hardness. This is primarily due to the fact that different instruments in the hands of different users give different readings. Much study has been given the subject in the Society but there is still a great need for a reliable portable instrument that will give uniform duplicable results.

TABLE I.—COMPRESSION-SET TEST RESULTS BY METHODS A AND B OF A.S.T.M. METHOD (D 395 – 36 T)[a].

Sample	A.S.T.M. Hardness Number	Compression Set, per cent	
		Constant Load	Constant Deflection
A....................	76	7.8	4.3
B....................	58	14.3	7.5
C....................	65	29.7	16.3
D....................	79	38.3	18.7

[a] Tentative Method of Test for Compression Set of Vulcanized Rubber (D 395 – 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I (1936); also 1936 Book of A.S.T.M. Tentative Standards.

Compression Set Tests:

Compression-set tests are important as an aid in the choice of the proper compound for engine mountings. For example, as shown in Table I, it is possible to have several compounds of the same hardness but capable of taking different degrees of permanent set under constant load or constant deflection. Experience has shown that compounds having a compression set higher than 25 per cent when determined by either method under A.S.T.M. Tentative Method of Test D 395 – 36 T are not satisfactory for motor mountings. Compression-set tests are used to measure the ability of a rubber compound to return to its original thickness after having been held in compression for a given period of time under definite conditions of load and temperature. They have their counterpart in actual use. Engine mountings are generally used in compression, and it is important that the rubber should not take too great a permanent set under this constant load. In general, a compound with a low permanent set is also more efficient than one with a high permanent set and therefore its effective life and usefulness will be much greater.

The test is made on samples of rubber compound cut from slabs $\frac{1}{2}$ in. thick prepared for the purpose or on samples cut from actual parts whenever possible. Two methods are provided. Method A is a test for compression set under a constant load, such as 400 lb. per sq. in., and Method B is a test for compression set under constant deflection. The choice of the method is optional but consideration should be given to the nature of the service for which correlation of test results may be sought. The standard sample comprises a disk of rubber $\frac{1}{2}$ in. thick cut with a circular die having an inside diameter of 1.129 in ±0.001 in. This gives a sample of 1 sq. in. in area. Under the constant-load method the sample is put in a clamp provided with a suitable spring which exerts a constant load of 400 lb. throughout the testing period of 22 hr. at 158 F. Under the constant-deflection method the sample is held at a constant deflection for the period of test. The amount of deflection varies with the hardness as follows:

A.S.T.M. HARDNESS NUMBER (3-LB. WEIGHT)	COMPRESSION OF RUBBER, PER CENT
130 to 71	40
70 to 36	30
35 to 12	20

Sometimes the size of the sample and load are changed from that shown in the A.S.T.M. method to make tests on the product possible. This test is strictly a laboratory test and is very useful as a control of the uniformity of the rubber and state of vulcanization. It is usually a part of every motor-mounting specification.

Metal-Adhesion Tests:

Most of the engine mountings in use today are of the metal-adhesion type. A laboratory test which measures the degree of adhesion to the metal is very important. The usual method of testing is to mount the actual part in a suitable tension machine in such a way as to pull the metal parts away from the rubber under a constant speed of jaw separation, at the same time recording the load in pounds required to break the rubber. This, strictly speaking, is the breaking strength of the part under tension or shear as the case may be. This value is sometimes converted to a value in pounds per square inch based on the contact area of the metal part and is a measure of adhesion in cases where the rubber separates clean from the metal. However, the degree of adhesion being obtained in most cases is such that the rubber will tear or break before separation occurs and, therefore, the value obtained is the breaking strength of the part.

As a control over the compound used and its uniformity in giving adhesion, the Society has published a Tentative Method of Test for Adhesion of Vulcanized Rubber to Metal (D 429 – 35 T).[3] This is a laboratory test and is useful for determining the uniformity of plating and stocks by using specially designed test pieces that can be plated and vulcanized to any rubber compound. This test can be used to predict the degree of adhesion a chosen compound will give.

In most laboratories, however, the actual parts are mounted in a suitable test machine, and the load applied at a constant speed until the part is broken. The load at which the rubber separates from the metal or at which the rubber breaks is considered the breaking strength of the part. Some engineers divide this value by the cross-sectional area of one of the metal parts and obtain a value in pounds per square inch. This value is not correct, since the cross-sectional area of the rubber is large and constantly changing during the pull. In many cases a small nick in the rubber will cause it to tear. The author therefore prefers to record only the breaking strength of the part as a whole and observe whether the rubber separated from the metal or whether the break occurred in the rubber.

A discussion on the testing of rubber must include the well-known tests for tensile strength, modulus of elasticity, and elongation at break before and after aging. These tests are strictly laboratory tests made on thin slabs prepared for that purpose. They play very little part in choosing a compound for a particular purpose. They do serve, however, to describe the quality of the compound chosen in terms that can be measured. Certainly a specification calling for 3500 lb. per sq. in. tensile strength requires a compound of much higher quality than one specifying 1500 lb. per sq. in. or even 2500 lb. per sq. in. They can also be used to control the uniformity of the compound. Tensile strength, elongation, and hardness are values that can be determined on both normal and aged compounds and when used intelligently place very definite limitations upon the quality of the compound and the compounding pigments used to make it. On the other hand the buyer of the rubber must not be misled into specifying values that will place such limitations on him that he is forced to pay for a higher quality than is actually needed.

Tensile strength and elongation in many cases can be determined on test specimens cut from the actual part and buffed to the proper thickness. Proper allowance must be made for variations that occur due to the cutting and buffing operation. Tensile strength values will invariably be lower and vary more on test specimens cut from actual parts than is the case where slabs 0.07 in. thick are cured for the purpose. When specifying tensile strength, therefore, the specification should state whether the test strips are to be cut from the part or whether slabs of stock are to be cured for that purpose.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1180 (1935); also 1935 Book of A.S.T.M. Tentative Standards, p. 1204.

Laboratory Flexing Tests:

In developing compounds suitable for use as engine mountings it is not always possible to subject them to expensive road tests in cars. A laboratory test similar to a service condition is, therefore, used. Since the action on an engine mounting in service is essentially rapid vibration under small deflections, a flexing test under compression is used to determine the relative life or efficiency of compounds. Abbott[4] first described this type of test, whereby a block of rubber of suitable size is flexed rapidly with a rotary motion while held under compression. Other tests suitable to actual parts are also used, the part being mounted in such a way that it can be flexed rapidly to simulate the flexing it receives in the car, and the number of flexes or hours to cause failure are recorded. The failure may be separation from the metal, or fatigue and blowout of the rubber.

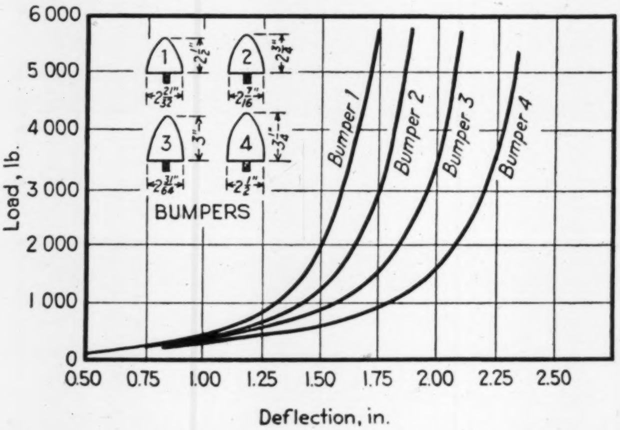

FIG. 5.—Load-Deflection Curves of Axle Bumpers Shown in Insert.

AXLE BUMPERS

Axle bumpers have in recent years become a rather important item on the car. Because of softer springs they come into action frequently. The usual laboratory tests used to evaluate bumper stocks are: load-deflection data, hardness, permanent set, and resilience and ability to withstand repeated shock.

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[4] F. D. Abbott, "Machine for Testing Rubber Products Used to Absorb Vibration," *Industrial and Engineering Chemistry,* Vol. 20, pp. 853–857 (1928).

The laboratory load-deflection curves on various compounds are determined as described under the discussion of engine mountings. These data, the hardnesses of suitable compounds, and the allowable deflection of the car together with the weight or force striking the bumper, are used in choosing the proper rubber compound. Load-deflection tests on several shapes of bumpers give data on the build-up rate of various designs. Figure 5 shows typical load-deflection curves for conical-shaped axle bumpers of heights varying from $2\frac{1}{2}$ to $3\frac{1}{4}$ in., as shown.

The quality of compound for bumpers need not be as high as for engine mountings. However, although the requirements for hardness, compression set, and load-carrying capacity might be as high as for engine mountings, the lower quality may be indicated by specifying lower tensile strength values.

As the failure of bumpers in service is usually the result of repeated shock, the laboratory impact test is valuable. The usual procedure is to mount the bumper in a fixed position and by means of a sliding arm driven by a motor repeatedly strike the bumper and compress it 50 to 75 per cent at a relatively slow rate of speed—say 60 times per minute. A record is kept of the number of impacts before failure occurs. Since the rubber bulges when compressed, the failure will usually show up as a rupture in the outer wall of the rubber at the point of greatest stress.

FAN BELTS

A fan belt is an entirely different type of product. Usually it is a structure involving a combination of fabric and rubber. The best test for evaluating its quality is to subject it to actual service conditions on a car. This, however, is a long-time test. Fan belt testing serves as a typical example of the usefulness of accelerated laboratory tests. The testing equipment usually consists of a series of pulleys, either two, three or four depending on the particular test being made. One pulley is driven by a motor and the others are connected either to a generator or to water brakes to supply the load. A belt is mounted and run until failure occurs. By this means the belt is tested as a complete structure. The test is accelerated by using higher speeds and higher loads than those used in actual service. Furthermore, the test is continuous.

Laboratory tests on the rubber compounds used in the belt are used for comparing one stock with another and are of some limited value. Flexing tests on the compounds themselves do not seem to be of much value. In the development of belts, therefore, reliance is almost always placed on the accelerated laboratory type of service test which is so designed that belt failure can be obtained in from 60 to 100 hr. It has been found from experience that there is a relationship between the hours to obtain failure in the laboratory and service life on the automobile. A belt which runs

60 hr. on a particular laboratory set-up will last longer in service than one which runs only 30 hr. The service life, however, will not necessarily be twice as long, because other factors, such as the effect of grease and oil, disalignment of the pulleys, etc., affect service life in the car.

Hardness, tensile strength, and friction-adhesion tests are used on belt compounds as control tests to determine the uniformity of the compounds being used and to control the state of vulcanization. At the same time belts are taken from production frequently and tested on the fan belt machines since a specification usually specifies the minimum number of hours a satisfactory belt should run under the particular type of set up.

OTHER PRODUCTS

There are many other rubber parts used on the modern car, including rubber-covered running boards, floor mats, sponge rubber, grommets, and gaskets. The laboratory tests used to determine and control their quality include those already mentioned. Running boards are tested for tensile strength, elongation, hardness, and adhesion to metal both before and after accelerated aging, the accelerated aging being carried out either in the air oven at atmospheric pressure and 158 F. or in the oxygen bomb at 158 F. with oxygen at 300 lb. per sq. in. pressure, or in the air bomb. Floor mats are usually tested for tensile strength, elongation, tear resistance, cracking when bent, and aging.

With the advent of the synthetic oil-resisting substitutes for rubber and their use in the modern automobile for gaskets and flexible gasoline connections, another test has come into prominence. That is the laboratory determination of the resistance of various compounds to the action of gasoline, oil, and water. No standard method has yet been developed for determining these qualities, but the Society's Committee D-11 on Rubber Products now has a subcommittee studying this problem. In the meantime, each laboratory has adopted methods more or less along the same lines. In general the method is to measure the volume of a suitable sample before and after immersing it in the desired liquid for a given period of time and at an elevated temperature. For example, one method involves immersing a sample 1 by 2 by $\frac{1}{4}$ in. in the desired liquid for 4 days at 158 F. The volume is measured before and after immersion by weighing in air and water. Another method such as used on radiator hose and heater hose consists of immersing 5-in. sections of the entire hose in water or anti-freeze solution for 5 days at boiling temperatures. The volume is measured before and after treatment by determining the volumetric water displacement. Instead of measuring the volume changes of rubber samples, some laboratories measure only the increase in thickness of the samples after treatment. This method usually gives inaccurate results. In addition to the determination of increase in volume, the sample is examined manually for its loss in hardness, tear resistance, and resistance to abrasion. These tests are valuable and to a certain degree give the desired information. It is hoped that very soon a suitable standard procedure can be provided.

CONCLUSION

Although the author has not gone into a great amount of detail in discussing the various tests applied to rubber products, it is hoped that this paper brings to light the importance of the various testing methods in evaluating rubber products. In addition, it is hoped that a realization of the importance of these tests will lead to continued efforts by Committee D-11 to write new and improved methods of test procedures.

DISCUSSION

Mr. Arthur W. Carpenter.[1]—The curve (Fig. 4) which Mr. Allen has shown, correlating indentation hardness readings using the Shore durometer with similar tests by the A.S.T.M. instrument, is interesting and very useful but additional emphasis may well be given to the fact, which he was careful to mention, that it represents only average conditions. In other words, the comparison should be regarded as approximate and should not be used as a basis for conversion of one reading to the other when precise data are required as in the enforcement of specifications. Similar curves may be prepared relating various other indentation hardness testing instruments as, for example, the Pusey and Jones plastometer, the Firestone penetrometer and the Adams densimeter. In most cases, however, considerable judgment is required in the use of such curves since the actual test conditions differ in the several methods, introducing substantial divergence from the curves when various types of rubber compounds are involved.

Of the five instruments mentioned, the A.S.T.M. hardness tester and the Pusey and Jones plastometer employ dead-weights to actuate their indenting elements. The other three use springs. Also, the sizes and shapes of the indenting elements as well as the magnitude of the operating pressures in the various instruments differ except with the A.S.T.M. and Firestone methods. In using the plastometer, two observations are necessary to measure the indentation while one reading suffices in the other cases since all except the plastometer have a pressure foot around the indenting element for establishing a surface plane to which the indentation is referred. The amount of pressure on this reference plane is not the same in the different methods, and, particularly with the hand-operated spring instruments, may vary widely with different operators. Spring loading is open to the objections that springs are subject to fatigue under constant use, giving incorrect readings, and that the magnitude of the applied load necessarily changes slightly according to the depth of the indentation.

In addition to these variations, there is another serious difference between the use of the dead-weight and the spring types of instruments which renders impractical the employment of correlation curves except when merely approximate information will suffice. With the spring type, the indentation reading is generally taken by applying the instrument to the rubber surface and observing the indentation *immediately*. This is particularly true of the testers held by hand where uniform prolonged application would be impossible. With the dead-load instruments, however, the reading is taken *at a definite time interval* after the application of the pressure to the indenting element. This period is usually at least 30 sec. and often 60 sec. or longer. Under these conditions, an immediate indentation occurs when the pressure is first applied, followed at once by a fairly rapid drift of the indicator which becomes progressively slower until no further deformation is shown. In ordinary operation, a reliable observation can hardly be made until a condition of equilibrium is reached at the end of a specified time. The same effect may be noted in prolonged contact with the spring instruments. This behavior suggests that the indentation consists of two portions, one of which results from elastic deformation and the other from plastic flow. An elastic deformation would occur instantly, and recovery upon release of the deforming force would be immediate and complete. Indentation due to plastic flow would be less rapid and the recovery, if occurring at all, would be slow and due only to the so-called "elastic after-effect." It would be expected, therefore, that plastic indentation would be particularly pronounced with rubber stocks having high permanent set. In practice, it is with this class of compounds especially, that correlation curves between the two types of instruments are found to be most unreliable. The usual delayed readings of dead-weight instruments appear, therefore, to include both elastic and plastic indentation while immediate readings with the spring devices show mainly elastic deformation. Unfortunately, ordinary rubber compounds are never perfectly elastic but always exhibit plastic properties to some slight degree so that the discrepancies mentioned are always present even though their magnitude may not be sufficient to be troublesome.

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[1] Manager of Testing Laboratories, The B. F. Goodrich Co., Akron, Ohio.

Mr. Allen mentioned that a small, convenient, portable hardness test instrument employing dead-weight loading is desired and needed. This suggestion is very much to the point and deserves serious consideration. However, it is generally appreciated that the development of an entirely satisfactory hardness instrument and test method is a very difficult problem because of the many requirements imposed by the various interests involved and the wide differences of opinion concerning precisely what is to be measured. It is unfortunate that so important a test for rubber as the determination of hardness has not been given more exhaustive study. The property of hardness is without question exceedingly complex and as yet, I believe, has not in a purely physical sense been adequately nor rigidly defined. Until that is done and agreement is reached, a completely rational solution of the hardness test problem appears remote. More concentrated effort than has been evident in the past would seem to be justified in view of the importance of the determination since this is one of the few tests which is rapid, generally applicable, and non-destructive. The existing methods are frequently used in specifications as well as for production inspection or uniformity control and because of their present unsatisfactory state they are sometimes seriously abused and misconstrued, especially in specification work. It is sincerely hoped that Mr. Allen's suggestion may stimulate further effort toward complete solution of the hardness test problem.

MR. C. H. ZIEME.[2]—I should like to add to Mr. Carpenter's remarks regarding the use of the Shore durometer for measuring hardness of rubber. It is a convenient portable instrument but has the disadvantage of variability in the hands of different operators due to their inability to apply the same pressure manually against the test specimen. This was illustrated in our laboratory when a number of routine operators experienced in the use of a durometer were asked to check the hardness of a number of control slabs of rubber having different degrees of hardness which were placed upon the platform of a scale. The pressure exerted varied from 2 to 11 lb. and obviously there was quite a variation in the individual hardness determinations. Since the personal element is such a determining factor in the uniformity of results obtained with a Shore durometer, this condition was improved considerably by mounting the instrument on a stand with a stationary platform and a movable attachment for the durometer provided with a 2000-g. weight to supply the pressure. While the durometer operating under a constant load is not as accurate as the A.S.T.M. hardness tester, it is being used successfully for control work and results obtained by different operators are fairly constant.

Through constant use, the spring weakens and it is, therefore, necessary to check frequently against standard control slabs of rubber. Where a number of instruments are used it is desirable to have a master instrument which is used only for checking and calibration.

MR. A. T. MCPHERSON.[3]—The McBurney indentation test[4] for asphalt tile has excellent possibilities for use in measuring the hardness of rubber because it takes the time factor into account. In this test the indentation is measured at two intervals, say 10 sec. and 1 min., after the application of the load, and the results are used to establish a time-indentation equation. This equation is logarithmic in form and has two constants which are highly characteristic of the material. From this equation it is possible to predict the indentation over long periods of time and to obtain results in good agreement with observation. The form of the instrument which was developed for asphalt tile is not very well adapted to soft rubber but it could probably be made suitable by changing the load and the indenting tool.

MR. ZIEME.—I should like to ask whether the author has given any consideration to the sun checking of rubber that is exposed on the outside of automobiles, such as sealing strips, and glass runs.

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[2] Service Engineer, The Republic Rubber Co., Youngstown, Ohio.
[3] Chief, Rubber Section, National Bureau of Standards, Washington, D. C.
[4] J. W. McBurney, "Indentation of Asphalt Tile," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 591 (1934).

MR. J. J. ALLEN.[5]—Sun checking tests are being included in specifications for rubber parts that are exposed to sunlight. The usual procedure is to cure rubber samples into test slabs or to use parts in the form they are used on the car. Suitable sized sections are then nailed on a board with a stretch of 100 per cent. These samples are then exposed to sunlight or to artificial ultraviolet light. The period of time elapsed before checks or cracks show up is recorded in some cases, and in others the amount of checking is noted after different periods of exposure.

The ultraviolet light method is more rapid, requiring in some instances only an hour or so. It has a disadvantage in that unless proper care is taken, erroneous results are obtained. Different sources of light give different results; heat effects must be taken into consideration. Quite frequently the results obtained with the ultraviolet light do not agree with results in natural sunlight.

MR. CARPENTER.—As regards tests for sun checking, I should like to mention that important variables requiring control in any artificial irradiation test are the variety and relative intensity of wave lengths of the particular light employed. Some years ago, considerable work was done on this question by Williams, Jecusco and others[6] but, even yet, sun checking appears to be an exceedingly complicated matter concerning which much remains to be learned. Nevertheless, artificial exposure methods involving irradiation under arc lights and various other lamps are being fairly widely used for purchase specification purposes, and shipments are sometimes accepted or rejected on the basis of tests which, though open to question, doubtless have to be employed for want of better methods. I believe that the Society's Committee D-11 on Rubber Products would do well to give consideration to this subject.

Since the dates of the work mentioned, there have been substantial improvements in the lamps that are available for exposure tests and much better control of the precise quality of light may now be obtained. This should be a very fertile field for investigation and one where standardization work would be of great service.

MR. J. P. COE.[7]—I should like to ask Mr. Allen whether he uses a test for measuring hysteresis or elasticity or the development of heat or any similar test for blocks which are to be used under compression load.

MR. ALLEN.—Brief reference is made in the paper to a flexing test on rubber blocks under compression, used to determine the relative life or efficiency of the compounds. The test was first described by Abbott and consists of a machine whereby a block of rubber is mounted on a plate which can be rotated off center at a high rate of speed. The block is held under compression with a load of 200 to 400 lb., depending on the hardness. The temperature of the rubber is usually measured after a period of time. In some cases the block is run until blowout occurs.

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[5] Chief Chemist, Mechanical Rubber Goods Division, The Firestone Tire and Rubber Co., Akron, Ohio.

[6] Ira Williams, "Oxidation of Rubber Exposed to Light," *Industrial and Engineering Chemistry*, Vol. 18, No. 4, April, 1926, p. 367.

F. P. Jecusco, "The Addition of Light to Accelerated Aging," *Industrial and Engineering Chemistry*, Vol. 18, No. 4, April, 1926, p. 420.

[7] General Sales Manager, Naugatuck Chemical Co., New York City.

Other types of machines have been described in the literature which involve impact tests with a pendulum as a means of measuring efficiency. A considerable amount of work is being done on methods of measuring efficiency of rubber compounds.

THE RELATION BETWEEN LABORATORY TESTS AND SERVICE LIFE OF RUBBER HOSE AND BELTING

By W. L. Smith[1] and Arthur W. Carpenter[1]

Synopsis

The paper discusses the various classes of laboratory tests employed in the evaluation of rubber hose and belting, with consideration particularly of the significance of the tests. Distinction is made between tests which are adapted to the control of quality uniformity and those which serve for estimating probable service life. The former are better suited to use in acceptance specifications since they are more thoroughly standardized, possess satisfactory reproducibility and are capable of yielding definite results which may be compared with established tolerances. The discussion covers individually in detail the usual quality tests for both the component parts and the assembled constructions of hose and belting. The use of performance tests for estimating serviceability is next considered. Mention is made of some of the important factors involved in design of these methods and the subject is developed by means of a few typical examples including a laboratory service test for pneumatic tool hose and dynamometer tests for belting.

Data are presented showing how quality tests frequently fail to correlate with service life and also how erroneous conclusions are sometimes drawn from performance tests when adequate consideration is not given to all of the variables in both the test and the service. However, the paper establishes the fact that, when properly designed and interpreted, laboratory performance tests may be used successfully for comparing service value of different specimens of rubber hose or belting in a particular set of conditions or for estimating probable useful life when an unknown specimen is compared with one for which the actual field performance is known. These tests are therefore extremely valuable in research and development work even though they are usually not sufficiently precise nor as yet well enough standardized for specification purposes.

Introduction

In the early days of rubber manufacture, adequate methods and suitable facilities for laboratory testing were unknown. An entirely new industry was being started. Production processes and equipment as well as new products, designs, compounds, and applications to service absorbed the attention of the great pioneers such as Thomas Hancock, McIntosh, Goodyear, Chaffee, and others.

In his personal narrative on "The Origin and Progress of the Caoutchouc or India-Rubber Manufacture in England," published in 1857, Hancock mentions the manufacture of what were possibly the first pieces of rubber hose and belting. He states, "In the early part of 1822, I began to make tubing of the cut sheet rubber, and afterwards with alternate plies of cloth coated with solution." Later he continues, "The cut sheet rubber began about this time, 1825, to be used for a number of surgical purposes, as before mentioned. All these kinds of articles were taken up by my late brother, John Hancock, and manufactured by him from materials supplied by me. . . . He also devoted a great deal of his attention to the manufacture of tubing, made sometimes solely of sheet-rubber, and also by uniting plies of cloth. This gradually led to the introduction of rubber hose-pipe, which met with a vast amount of opposition from the leather-hose makers, and amongst brewers and distillers—particularly the men who were in league with the old makers. It appears that the leather hose used in breweries could not be made so perfect as to prevent a great loss of liquor. It was stated to one of the great firms (Messrs. Barclay and Co.) that the rubber hose would not admit of any escape; and one of the firm insisted upon its being tried: it was found effectual, but imparted a bad flavour to the liquor. This was for a time a great obstruction to its use; but it was found that, by allowing waste liquor to run through the hose for a while, it became sweetened and ultimately (if I mistake not) about 1800 feet was constantly in use in that brewery alone. Others followed, and rubber hose and tubing became a staple manufacture, and continues so to this day. The hose-pipe was composed of two, three, or more plies of cloth, coated on both sides with solution, and then rolled up on mandrels. They were generally lined with sheet rubber; suction hose had spiral wire inside to prevent it from collapsing."

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[1] Assistant Technical Superintendent, Mechanical Goods Division, and Manager of Testing Laboratories, respectively, The B. F. Goodrich Co., Akron, Ohio.

In 1825, Hancock obtained a patent for a method of manufacturing artificial leather employing a solution of rubber for combining fibrous substances such as wool, silk, cotton, hemp, flax or hair in their manufactured or unmanufactured state. In discussing this, Hancock says, "I also united as many plies of cotton, linen or woollen cloth together as would make up the thickness I required. I also introduced into the solution, for some of the most common purposes, black resin, size, glue, ochre, powdered pumice, whiting, etc. That made of a number of plies of cloth was used during this year, 1825, for the backs of cards in carding machines, instead of leather. . . . This article is also used extensively as printer's blankets in calico-printing. I also made in this manner very strong straps for driving machinery, and furnished one of the first to the late Sir Isambert Brunel, for his engine, when sinking the shaft for the Thames Tunnel. Such straps have continued ever since, and are still, much used." Thus, the manufacture of rubber hose and belting preceded by some fifteen years the discovery of vulcanization.

The earliest reports of any quantitative laboratory tests of the physical properties of rubber were made following 1851, more than a quarter of a century later, when Hovine, Debonnefoy, Boileau, and others, described their compression experiments. In 1869, Villari reported his studies on the relations between tensile stresses and the resulting strains. This test work, however, dealt principally with the measurement of the properties of the rubber material itself rather than with the behavior of complex rubber products, and laboratory testing of rubber articles as now carried out was practically non-existent until nearly the close of the nineteenth century. During all of the intervening period, hose, belting, and the countless other rubber articles which came into use, were evaluated by trial in actual service. Consequently, progress was slow, material in test was frequently lost, and sometimes results were none too reliable because of uncontrolled and unknown variations in the operating conditions. Present physical laboratory methods have gone far to overcome these difficulties, yet even today the final and conclusive judgment of the service value of rubber products rests in the last analysis on their actual performance.

CLASSES OF TESTS

The tests usually made on rubber products may, of course, be divided into those involving chemical analysis and those which depend on physical measurements. The former were relied upon, exclusively, prior to the development of adequate physical methods. Obviously, chemical analysis is useful for determining and controlling the composition of the material making up a rubber article. The composition, however, may have very little bearing on the fitness of articles for use and rubber compounds having the same composition may often vary widely in physical properties depending on the methods and treatment during manufacture. Though still sometimes used as a means of evaluating quality of rubber products, chemical tests are now generally regarded with much less favor than physical measurements which usually are more directly related to the actual service and give results more rapidly. In this discussion, therefore, consideration will be given only to mechanical rather than chemical methods.

Mechanical tests of rubber products are often for convenience grouped in two classes: the first embracing those intended to measure fundamental physical properties such as dimensions, specific gravity, the stress-strain relationship in compression or tension, hardness, permanent set, color and the like; and the second, performance tests, including tests for aging, adhesion, resistance to abrasive wear, tearing and cutting, fatigue tests of various kinds as well as direct imitations of actual service under laboratory conditions. Tests of the performance type are more suitable for research and development purposes than for use in purchase-acceptance specifications because they are frequently quite difficult to control in such a way that the service variables are reliably duplicated. Often the results secured are not sufficiently precise with respect to reproducibility nor definite enough in "end point" to be satisfactory in specification work. Tests of the first class, however, have been in use for a much longer time and have been subjected to more study and standardization since they were the earliest physical tests to be developed. Their results are usually definite and are expressed in terms generally understood and accepted. They are, therefore, valuable for checking product uniformity and are adapted to use in specifications though they may fail to give a true indication of actual service life.

This classification cannot be regarded as absolutely rigid since exceptions occur in each group. Some particular physical property may govern the fitness of an article for service in which case its measurement would have performance significance. For example a measurement of breaking strength may have considerable bearing upon the ultimate success of a product in service even though normally the article would not encounter stresses of so great magnitude. Also, certain performance tests possess some of the characteristics of the measurements of physical properties. Thus, tests for aging, adhesion and a few special tests such as behavior under hydrostatic pressure or in chemicals and solvents have been found useful for control of both service quality and product uniformity. A distinction must therefore be recognized among performance tests between those which are well established and capable of giving accurate, duplicable results, and the other laboratory service tests, which like the many attempts to imitate actual use are limited in precision and reproducibility but are valuable in development work for judging probable life of products.

In the discussion of laboratory testing of hose and belting, attention will be directed first to the methods usually employed in acceptance specifications for control of uniformity, and consideration will then be given to laboratory performance tests of the imitation service type. In both cases, it will be convenient to deal separately with the two groups of products. Owing to the innumerable styles, sizes and constructions of hose and belts manufactured for many widely different purposes, complete treatment of the subject obviously cannot here be given and it is necessary to limit the discussion to some typical instances to exemplify the relation between laboratory tests and service life.

SPECIFICATION TESTS

Specification Tests Employed for Hose:

A typical hose may be regarded as consisting essentially of an inner rubber tube, a carcass, and a cover. The tube serves as a flexible retaining member for the material inside the hose, resisting chemical action, abrasive wear, or heat. The carcass surrounding it is designed to provide adequate strength to withstand the working stresses and is comprised of plies of fabric, yarn, wire or other reinforcing material usually impregnated or embedded in rubber. The outer cover is also generally rubber, but manutured fabric, woven or braided yarns and other materials are used in some types of product. The cover is primarily an envelope protecting the structure from external deteriorating influences although in certain cases, like cotton-jacketed fire hose, it serves as the principal reinforcing member. This basic construction as outlined is capable of many variations in detail depending upon the methods employed in manufacture and the service requirements. In testing these varied products, some methods are of general applicability and since these procedures are fully given in the Society's Tentative Methods of Test for Rubber Hose—Braided Construction (D 379 – 33 T) and Rubber Hose—Wrapped Construction (D 380 – 33 T),[2] and in the standards for other materials, they need not be repeated here. It will suffice to point out that the complete test analysis of a given construction requires investigation of the properties of both the component parts which make up the final hose structure and of the finished product itself.

In evaluating the component parts of hose, tests of the rubber portions are limited to the tube and cover compounds since no satisfactory method is available for separating the rubber in the carcass from the fabric in such form as to permit adequate physical testing. Measurements of thickness, determinations of tensile strength and elongation or stress-strain properties, aging, permanent set, and sometimes special tests for resistance to water, oil, or chemicals, are commonly employed. The measurement of the thickness of the tube or cover taken from hose is complicated by the necessity of removing irregularities of surface by buffing when precise results are required as in specification work. The importance of uniform practice in this procedure is evident because of the relation of this requirement to the amount of material employed in manufacture which may affect both cost and serviceability. The tension tests are of value primarily for quality classification of the compounds and generally their relation to anticipated service is only indirect. The aging and special tests have more definite service significance. The test for permanent set adds very little to the information secured from the others and except in a few specialized cases is considered to have no relation whatever to the service value of the product.

Prior to manufacture, the other materials making up hose such as cotton fabric or yarn, wire and the like, are tested in the usual manner as specified by the Society for the particular materials. Such tests are essential for the design and development of hose constructions. It is difficult, however, to obtain satisfactory specimens from manufactured products although the rubber may be fairly well removed by the use of suitable volatile solvents. Experience has shown that the properties of cotton fabric, or other reinforcing materials taken from finished hose, will have been altered to some extent by the effects of the various processes of manufacture. For these reasons, the materials and construction of the carcass of hose are usually evaluated by hydrostatic pressure tests of the finished product.

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, pp. 909 and 915, respectively (1933); also 1935 Book of A.S.T.M. Tentative Standards, pp. 1243 and 1249, respectively.

The uniformity tests generally applicable to the whole hose structure and suitable for use in specifications are not very numerous. Included in these are measurements of dimensions, tests of adhesion between component parts, and hydraulic pressure tests. Though the significance of dimension measurements is obvious, the difficulties involved in their accurate determination on flexible articles made of soft materials, like many rubber compounds, are often not appreciated. Careful standardization of methods is essential if disagreements are to be avoided. Tests of adhesion aid in classifying and controlling carcass compounds and they also frequently have bearing on the serviceability of the hose since the entire structure must usually be adequately bonded together in order to secure satisfactory performance. However, ply separation may occur in use, as a result of flexing, heat, or other causes, under much smaller applied stress than that employed in the adhesion test. Therefore, hose showing the strongest adhesions may not necessarily always give maximum service life. A similar situation prevails with respect to hydraulic pressure tests. While very useful in controlling uniformity and extremely valuable in development of new constructions, measurements of bursting strength may fail to give reliable performance indications. An excellent example of this occurred with a type of water-spray hose of wrapped construction, made by a prominent manufacturer, which showed in test an average bursting strength more than three times the equivalent of the normal operating pressure. Yet, in nearly all cases, the hose failed in service far short of its anticipated life. This was simply a case where a test which truly evaluated a particular characteristic failed to take into account other conditions of use which were of greater importance. Sometimes, in such instances, modifications in hydraulic-pressure testing add valuable information. For instance, pressures far below the ultimate may be applied for extended periods and the effects on the dimensions observed, or the hose may be kinked or bent so as to change the stress distribution during the load application producing failure at lower pressures. Such variations are of little additional value for uniformity control of product but frequently they definitely improve the service significance of the test results. When a hose is designed for some certain specific purpose, specifications covering it may include special tests intended to evaluate dominant requirements of the service. An example is the use of immersion tests in solvents for hose which must resist their action. These special tests have generally not been well standardized and the obtaining of accurate, quantitative results by means of them is difficult. They are certainly of qualitative value however, and when several substantially different specimens are under consideration, these tests are especially useful as aids in the selection of the hose which is best adapted to the intended service.

Specification Tests Employed for Belts:

Rubber belts may be classified according to their use as those which serve for transmission of power in driving machinery and those which are used for transporting materials. Power transmission belts are of two general types: either flat bands, made up of plies of cotton duck or cord impregnated and strongly adhered together by rubber compounds; or continuous loops of trapezoidal or V-shape cross-section containing an inner structure of cotton fabric or cord embedded in rubber. These belts travel at rapid speeds over small pulleys and must possess a high degree of flexibility together with great strength and good friction properties on the pulleys. In order to meet these requirements they are made as thin as is consistent with strength and durability and their surfaces are generally of fabric with only a thin film of impregnating compound on the outside. The other class, which includes conveyor and elevator belting, resembles the flat transmission type in internal construction but differs in that heavier bodies and thick rubber covers may be used since speeds are slower, abrasive wear more severe and extreme flexibility less important because they are run over pulleys of relatively large diameter. In all cases, the necessary strength and stretch characteristics of rubber belts are primarily functions of the fabric structure rather than of the rubber although these may be profoundly influenced by the nature of the compound, its distribution in the belt and the manner in which it is applied.

TABLE I.—COMPARISON OF SOME TYPICAL LABORATORY TESTS.

Compound	Tensile Class, lb. per sq. in.	Index of Abrasion Resistance			Tearing Strength, Crescent Method, lb. per 0.1 in. thickness	
		DuPont Machine	New Jersey Zinc Co. Machine, Without Lifts	Goodrich Loose Abrasive	Longitudinal	Transverse
Reference standard	4400	100	100	100	75.9	78.9
Cover A	3500 to 4000	96.0	42.8	82.2	46.9	43.9
Cover B	2500 to 3000	72.0	48.2	61.2	45.0	43.0
Cover C	1400 to 2000	43.7	60.8	54.2	50.5	53.9

In checking the component parts of a belt, similar difficulties to those with hose are encountered. Tests of the rubber portions are limited to the thick covers of conveyor and elevator belts which may be readily removed and investigated for dimensions, stress-strain properties, permanent set, resistance to aging, tear and abrasive wear, as well as for the action of any chemical substances such as oils or fats, fruit juices, or the like. These tests are useful for control of uniformity of a single compound but as with hose, any implication as to service relations must be regarded skeptically in view of frequently occurring contradictory results like those in Table I obtained with three typical belt-cover compounds. Suitable specimens of the "frictioning" compounds for most physical tests cannot be secured from finished belts and reliance for their control must be placed on adhesion tests of the structure. In development work, fabric tests are of great importance for design and control of the strength and elastic properties of the product. For use by consumers, however, they are less satisfactory than the measurement of the actual stress-strain relations of the finished belt since the characteristics of the fabricated structure can seldom be determined by integration of tests of the individual parts. Fabric specimens from flat belts may be quite readily obtained by "degumming" but, as with hose, the original properties will have suffered change during manufacture. With V belts it is usually impractical to obtain any suitable test specimens of component parts by dissection or "degumming" and even if this were done, the results of the tests would be of little value as the service performance is so largely governed by structural design factors.

Tests of finished belting constructions for specification purposes involve principally measurements of adhesion values between the various parts and tensile strength, ultimate elongation and stress-strain determinations of the finished belts, as described in the A.S.T.M. Tentative Methods of Testing Rubber Belting Used for Power Transmission (D 378 - 33 T).[3] The same procedures may be used for conveyor and elevator belts if the thick covers, when present, are first removed. These tests are exceedingly valuable in the control of product uniformity and they often assist in judging serviceability. However, belts sometimes fail from ply separation even though in test they showed superior ply adhesion, and premature breaks in the fabric during use occur with belts which originally possessed satisfactory properties in tension tests. These limitations in service significance exist with the simple tests of separate properties because other influences acting on the product during use are not taken into account. The ultimate performance is governed by the resultant of all of the operating conditions instead of just certain ones, and knowledge of testing has not as yet reached the point where it is possible, so to speak, to synthesize the actual service relation from measurements of the individual properties. These specification tests therefore give indications as to probable performance only when the properties measured are predominant in governing the useful life, but this in no way detracts from the utility of the tests for control of uniformity.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 904 (1933); also 1935 Book of A.S.T.M. Tentative Standards, p. 1255.

A typical instance with belting where the specification tests failed to indicate service quality may be cited in the case of a coke conveyor on which rather complete information is available. The belts used were 42 in. wide and 500 ft. long, constructed of 8 plies of 32-oz. duck with a friction compound giving 16 to 19 lb. commercial ply adhesion. The covers were $\frac{1}{8}$ and $\frac{1}{4}$ in. of a standard type compound averaging 4000 lb. per sq. in. tensile strength with tie cloth included in both top and bottom. The normal service expectancy for similar belts based on actual records prior to 1930 in the same installation was the conveying of 2100 tons of coke per lineal foot of belt. Under ordinary conditions this amount of material would be carried in approximately 26 months. A belt placed in service in 1931 was recently examined. In 41 months of intermittent operation, only 1100 tons of material had been handled and the belt was cracked and in unsatisfactory condition. Examination of the manufacturing record and test data for this belt showed it to be essentially the same as the former ones on which the expectancy was based and no difficulties or changes could be found in the installation. However, in formulating the normal expectancy and in testing the belt quality and uniformity, no account was taken of the great difference in duration of the service in which only about half as much material was carried in nearly twice the time. Here, the governing factor in useful life was probably not the tonnage but time, during which moisture might penetrate the belt through cover burns or cuts and, with aging, cause deterioration incapable of being measured by present-day specification tests.

LABORATORY SERVICE TESTS

Since the older methods already discussed have so often been found deficient for evaluating the relative excellence of rubber hose and belts for their intended service, the trend of development in recent years has been toward increased use of performance tests of the imitation-service type. In these, it is aimed to duplicate as closely as possible in the laboratory the conditions of actual use and thus to secure data directly related to field performance. Unfortunately, this is not as easy as would at first be thought because very thorough analysis of both the service and the test are necessary and the test must usually be accelerated in order to obtain results in a minimum time. It is surprising how often essential factors are overlooked either because of lack of adequate definition of the service or because of improper design of the test. Acceleration of failure is usually accomplished by increasing the temperature, the severity and intensity of the forces acting on the sample, or the operating speeds. When the normal balance of service conditions is upset in this procedure, misleading results are almost certain to be obtained unless the emphasized variables are those principally responsible for failures during use. Therefore one criterion of a good laboratory service test is its ability to produce failures closely resembling those obtained in the field.

Tests of this class are frequently dynamic in that they may involve repeated stress application producing flexure, extension, compression or impact alternating with recovery. Often other variables such as internal pressure, static load, aging, action of gases, oils, chemicals or heat may be included simultaneously in various combinations. Sometimes, as when testing belts, the specimens are made to transmit power as well as being subjected to static load. These tests are practically never standard but are designed specially for each type of product in a particular application. They are not usually suitable for use in specifications both because they are expensive and because the results are not highly precise, requiring considerable discretion in their interpretation. However, when properly designed and interpreted, they give excellent service indications.

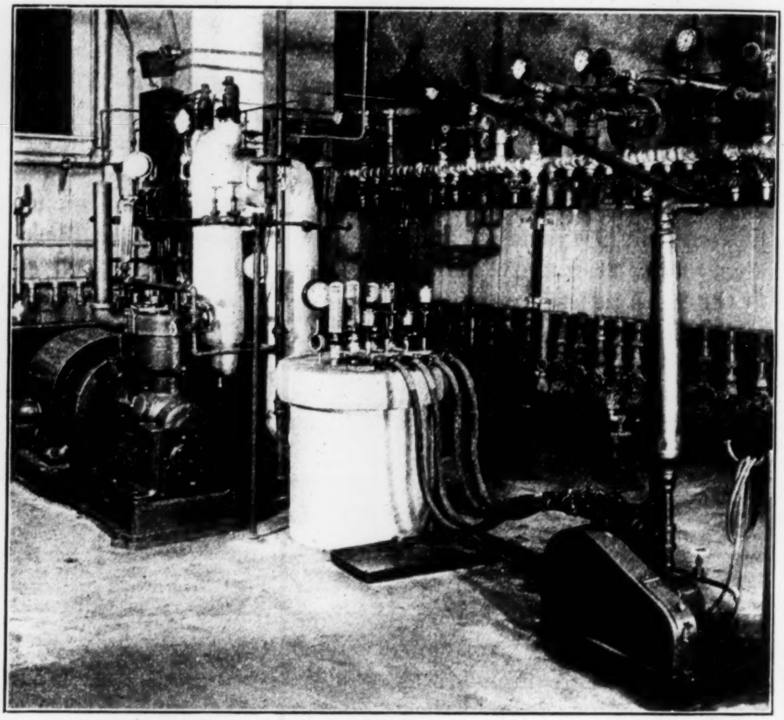


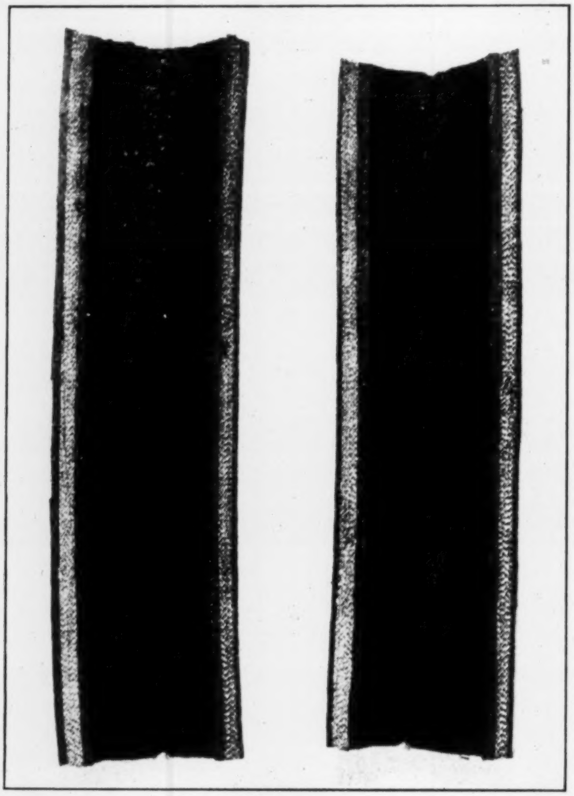
FIG. 1.—Apparatus for Testing Air Hose.

Hose Tests of Service Type:

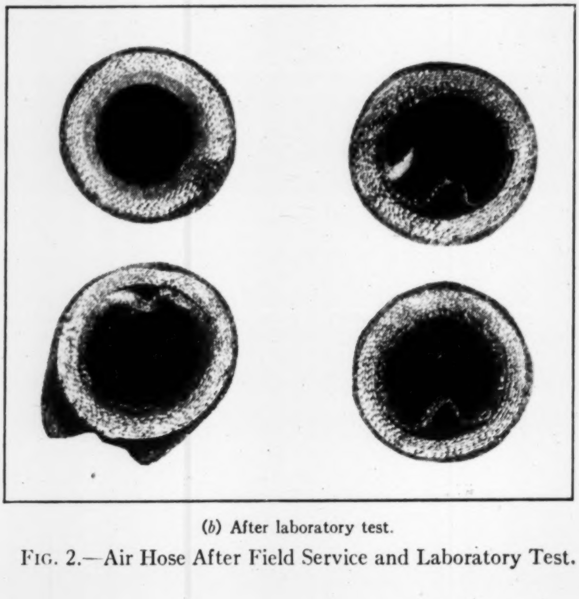
Laboratory service tests of hose are so numerous and varied in design that consideration may be given here to only a few typical examples. The test equipment for one which has been found useful in evaluating the performance of pneumatic tool hose is illustrated in Fig. 1. This example demonstrates in a representative way some of the principles which must be observed in this class of testing and shows the manner in which advantages are obtained and limitations encountered. The apparatus consists of an air compressor which discharges into a receiver through equipment arranged to give controllable constant temperature and pressure in the receiver. The compressed air from the receiver flows through the hose specimens in test after having received a known addition of lubricating oil at the receiver outlet. The compressed air is released from the hose through a valve mechanism which simulates the impulse action of a pneumatic hammer. Thus, the test includes as factors which are important in service (1) internal pressure with rapid impulses suddenly applied and released, (2) elevated temperature, and (3) action of oil used for internal lubrication of the tool. External influences, such as abrasive wear for example, are omitted. Four specimens connected to the receiver in parallel may be tested at the same time. Obviously, comparisons may readily be made between the four specimens which for valid results should all have the same length and internal diameter.

In this test the hose is considered to have failed when the tube leaks air as is evidenced by oil seepage through the carcass. Usually, the covers are removed from the specimens for about a foot at the receiver end to facilitate detection of this failure, which takes place in that region since the hose is there subjected to maximum temperature and oil deposition. The operating time until failure occurs is used as the basis for evaluation. Figures 2 (*a*) and (*b*) show specimens from both field and laboratory indicating that the types of failure are similar though the laboratory samples have suffered somewhat more from oil and less from hot air than the field samples. This is doubtless because these two deleterious factors were not in the same relative proportion during test as in use. Failures of this type have been quite usual in service but previous attempts to duplicate them in the usual laboratory tests had proven unsuccessful.

In order to obtain data which may be interpreted in terms of actual life instead of merely as a test comparison, it is necessary that one, at least, of the four specimens in a test shall be a sample of hose of which the field performance, in hours of service, is known. When the test life of such hose is compared with that of similar specimens having unknown service value, the probable life of the latter may be proportionally estimated. The values thus obtained are usually only approximate as service hours in the field can seldom be secured with much accuracy. To illustrate specifically, assume that hose operating a pneumatic hammer is connected to a compressor furnishing air at 80 lb. per sq. in. pressure and about 175 F. tem-



(*a*) After field service



(*b*) After laboratory test.

FIG. 2.—Air Hose After Field Service and Laboratory Test.

perature. The air carries a varying amount of oil and the tool strikes 400 blows per minute. From field records the hose is estimated to have an average service life of 1500 hr. In testing a sample of this hose in the laboratory, the failure might be hastened by using 100 lb. per sq. in. pressure of air delivered at 250 F., 12 drops of oil and 690 valve cycle impulses per minute. Under these more severe conditions the hose might fail in 150 hr. or in the ratio of 1 to 10 with the service life. A similar unevaluated specimen tested at the same time which lasts 200 hr. might reasonably be assumed to be capable of giving approximately 2000 hr. of service, especially if the test relation was confirmed by repeated checks. Experience over a period of years has demonstrated that, for development purposes, air hose constructions may, in this manner, be successfully evaluated in the laboratory when the service failure is of the particular type described. However, pneumatic tool hose sometimes fails from other factors than those here included. Figure 3 shows such a service where external abrasion is so severe that the hose may be worn and cut, causing it to burst before the inside deterioration has advanced sufficiently to be a serious contributing element. The test described is of little value in such a case and hope for improvement of product must depend on tests of resistance to abrasion, tearing and cutting until failure conforms to the proper type. Quite obviously, because of its approximate nature, the test described is not suitable for use in specifications for acceptance purposes.

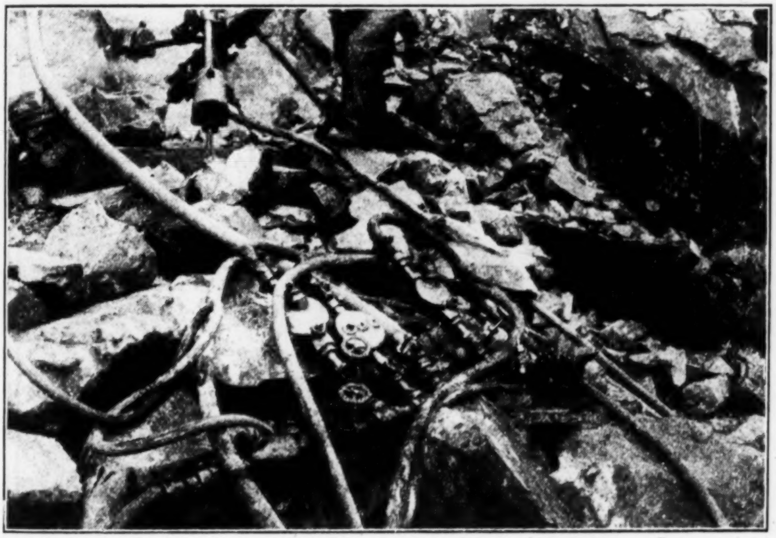


FIG. 3.—Abrasive Service for Air Hose.

Many other laboratory service tests have been employed for evaluation of different kinds of hose. Two interesting ones, used for steam hose and

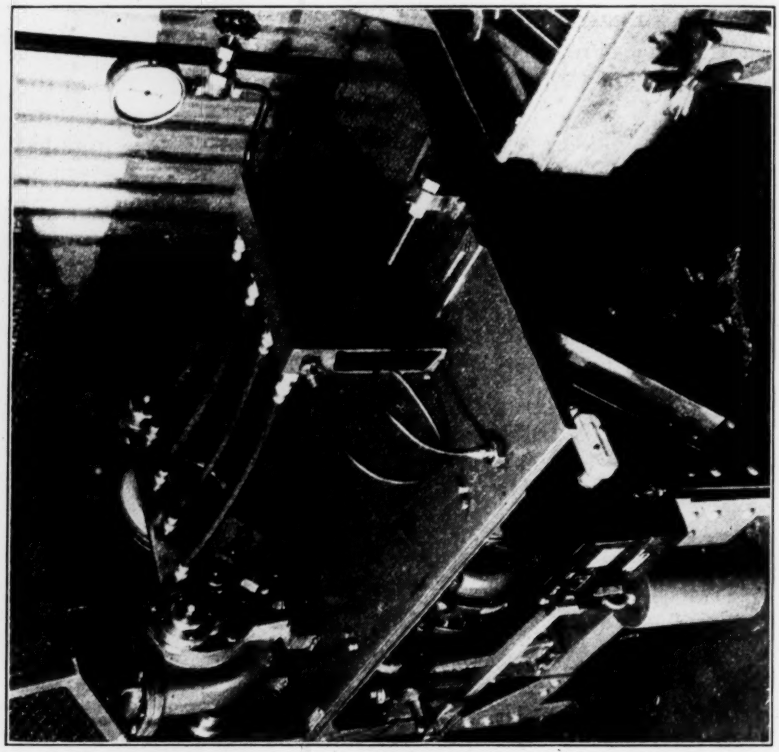


FIG. 5.—Hydraulic Brake Hose Performance Test.

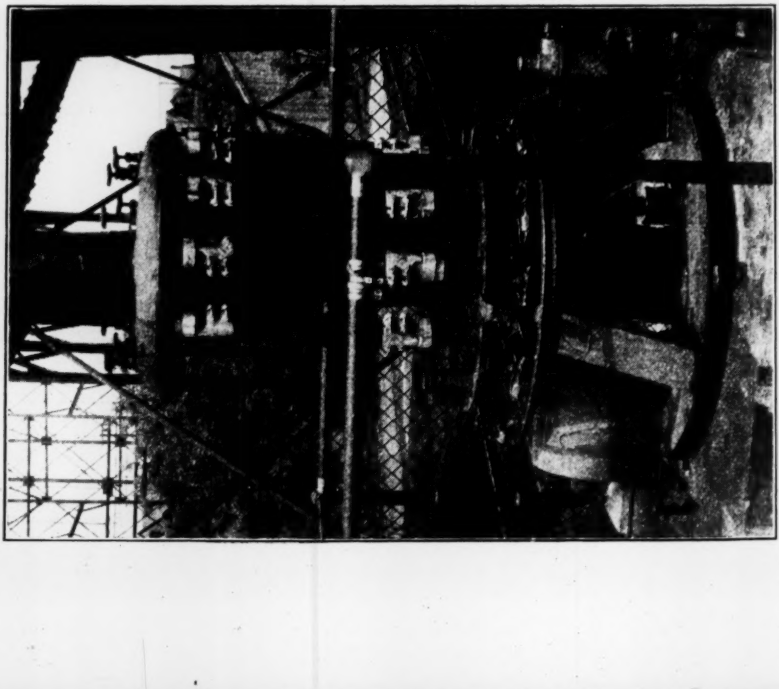


FIG. 4.—Steam Hose Performance Test.

hydraulic brake hose, are shown in Figs. 4 and 5, respectively. These employ different combinations of deteriorating factors than the air-hose test but the same general methods of analysis, design and interpretation are involved. Both have been very successfully employed in predicting service life and have aided greatly in the rapid development of improved products.

Belt Tests of Service Type:

The actual operating conditions of power transmission belts are often reproduced in the laboratory for test purposes by the use of motor-generator dynamometer sets as illustrated in Figs. 6 (*a*) and (*b*). With equipment of this sort, a wide range of belt thicknesses, speeds, pulley sizes, and tensions may be employed. Both the power input at the motor pulley and the output at the generator pulley may be controlled and measured, permitting calculation of the mechanical efficiency of the belt being tested. By suitable regulation of the power relations and the total tension, a known amount of creep and slip may be obtained. The details of the apparatus and methods of use have been discussed previously by Sturtevant[4] and others, and need not be repeated. It is sufficient to say that these dynamometers make possible, for power transmission belts of both the flat and V types, about as complete and satisfactory laboratory service tests as are known for any product. By suitable adaptation, valuable service information on conveyor belts may also be obtained. Dynamometers provide actual service under laboratory controlled conditions and the results are reliable indications of performance in installations where similar conditions exist.

Since motor-generator sets are expensive both to install and to operate and since the volume of test output is limited as only one belt is run at a time, other less complete service tests for belts have been developed. In one of these, described by Sturtevant, nine belts are driven under controlled tension from a line shaft and are loaded by means of Prony brakes. Other similar equipment employs water brakes for absorption of the transmitted power. Such apparatus is used exclusively for endurance life tests. Kimmich[5] has described a multiple unit test machine on which belts are run over pulleys with dead-weight tension but without power load. He developed a formula relating the hours of operating life required to produce ply separation, with pulley diameter, belt speed, number of plies and unit tension and showed that, with flat transmission belts, the effect of adding horsepower load is merely an adjustment of tension. Kimmich's work indicated that, with flat belts at least, the dead-load test would give similar endurance results before ply-separation, to those obtained with power

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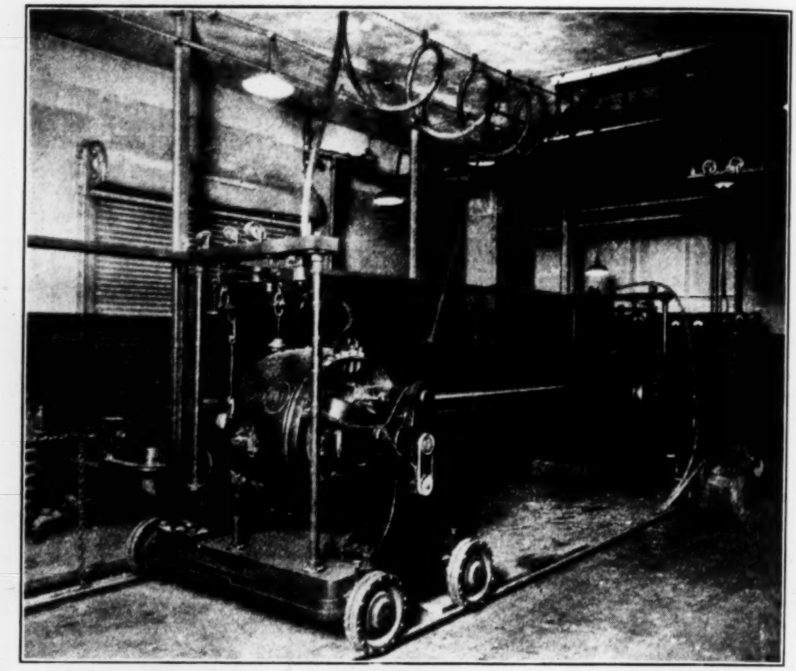
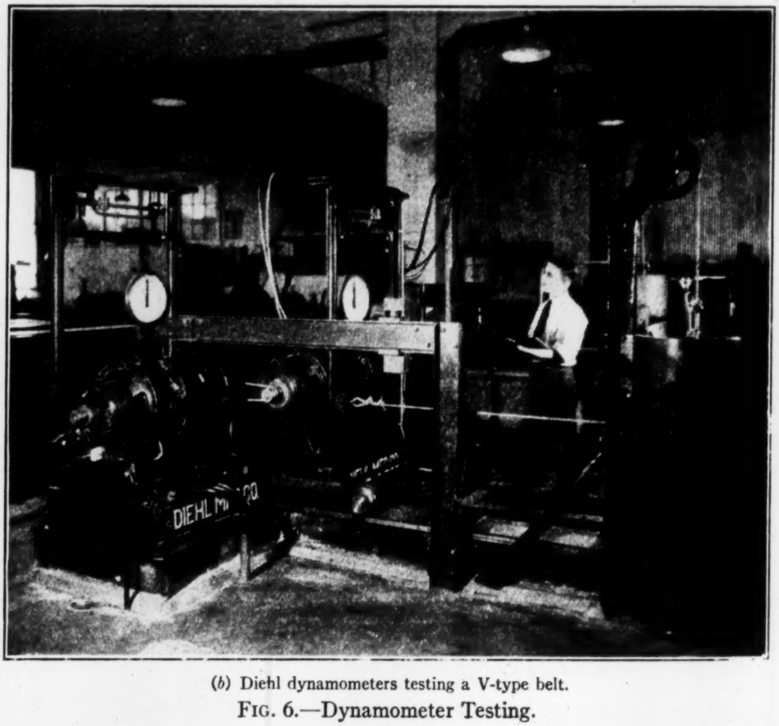
[4] W. L. Sturtevant, "Rubber Power Transmission Belting, III," *India Rubber World*, Vol. 83, No. 3, December 1930, p. 67.

[5] E. G. Kimmich, "Service Tests on Rubber Belts," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 957 (1930).



(*a*) Sprague dynamometers testing power transmission belting.



(*b*) Diehl dynamometers testing a V-type belt.

Fig. 6.—Dynamometer Testing.

loading if proper tensions are employed. This kind of test has been extensively used for both flat and V-type belts. Another laboratory method for evaluating ply-separation failure is the Scott flexing machine test described in the A.S.T.M. Tentative Methods of Test for Flexing of Rubber Products (D 430 – 35 T).[6] Sturtevant secured fair correlation between Scott flexing machine results and dynamometer endurance tests though neither agreed with the quality indications of ply adhesion measurements. The data in Table II where the dynamometer test was run without transmitted power fails to show similar correlation.

TABLE II.—COMPARISON OF LABORATORY TESTS OF FLAT TRANSMISSION BELTS.

BELT	PLY ADHESION, LB. PER IN. WIDTH[a]	SCOTT MACHINE, AVERAGE NUMBER OF FLEXURES[b]	DYNAMOMETER, ENDURANCE TEST, HR.[c]
A	26.7	60 000	87
B	24.2	48 000	153
C	24.0	25 500	250
D	22.9	56 800	208
E	17.8	9 000	15
F	15.8	8 000	54
G	14.0	20 000	20

[a] Tentative Methods of Test for Adhesion of Vulcanized Rubber (Friction Test) (D 413 – 35 T), Machine Method, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1173 (1935); also 1935 Book of A.S.T.M. Tentative Standards, p. 1197.

[b] Tentative Methods of Test for Flexing of Rubber Products (D 430 – 35 T), Method A, *loc. cit.*

[c] Belts, 30 ft. long, 6 in. wide, 4 plies, spliced with alligator fasteners. 100-hp. Sprague dynamometers, 4 in. diameter steel pulleys with $\frac{1}{8}$ in. per ft. crown. Belt speed, 1000 ft. per min. Total tension, 20 lb. per in. per ply.

TABLE III.—COMPARISON OF LABORATORY SERVICE TESTS OF V BELTS.

Type of Belt	Pulley Flexing Dead-Load Endurance[a]		High Speed Dynamometer with Power-Load[b]		Stretch at Failure, per cent
	Hours	Ratio to Type A	Hours	Ratio to Type A	
A	491	1	1.5	1	4.5
B	765	1.6	2.1	1.4	2.2
C	917	1.9	249.0	166.0	2.1

[a] 75 lb. total tensions; 3250 r.p.m.; drive sheave, $3\frac{1}{8}$-in. outside diameter; loading sheave, $6\frac{3}{8}$-in. outside diameter.

[b] Diehl dynamometers; 75 lb. total tension; 4000 r.p.m.; drive sheave, $7\frac{3}{8}$-in. outside diameter; loading sheave, 6-in. outside diameter; idler sheave, $3\frac{5}{8}$-in. outside diameter; horsepower load to give 2 to 5 per cent slip (approximately 8 hp.).

These simplified tests have been very helpful in development work but attempts to use them for complete service evaluation have often led to erroneous conclusions. This appears to be caused by placing too much emphasis on certain of the operating variables and not enough on others as a result of the acceleration of failure by heavy tensions, high speeds and small pulleys. For instance, one important factor which causes difficulty and which is not controlled in the accelerated laboratory tests is the effect of heat. Belts may be made strong to withstand heavy tension but when this decreases flexibility and increases operating temperature it is possible that the effect on service life may be indicated only in tests where transmitted power is included. An illustration is the comparison of test results of several types of V belts shown in Table III. Types A and B differed somewhat in construction but contained ordinary compounds of the sort long in use, while type C was specially designed to have high temperature-resistance and low heat-generating properties. If the dynamometer test is considered to represent a reliable measure of performance conditions, the inaccuracy of the service indication of the dead-weight test is obvious.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1186 (1935); also 1935 Book of A.S.T.M. Tentative Standards, p. 1229.

Even when laboratory tests of V belts include power load, their service indications may not be strictly proportional to the results in field performance. Data furnished by a motor-car manufacturer using a pulley test with power load shows that in 1930, V-type fan belts gave endurance life of 50 hr. In 1936 the figure had been increased to over 500 hr. in the same test indicating an improvement of 1000 per cent. Road tests, with duplicate belts, however, gave approximately 20,000 miles average service in 1930 and 29,000 miles in 1936 or an actual increase of only 45 per cent. While both tests demonstrated improvement of the belts, the magnitude of the change was not at all correctly shown in the laboratory.

CONCLUSION

The considerations here presented have led to the conclusion that it is often possible, in properly designed laboratory tests, to secure valuable indications as to service life of rubber hose and belts but such evaluation requires the use of tests of the performance type. Since these are for the most part comparative and of low precision, a background of actual service experience is essential for adequate interpretation of the test data. Laboratory performance tests are, however, extremely useful in product development and serve to eliminate from consideration articles radically unfit for use.

THE TESTING OF RUBBER INSULATED WIRES AND CABLES

By Dean Harvey[1]

One of the most important factors in the improvement in quality and in the standardization of rubber products has been the development of standard methods of test. These test methods have been used in research and for control of quality in manufacture, and by consumers in purchasing.

A significant feature in the testing of rubber insulation on wires and cables has been the trend toward performance specifications for purchasing rather than to specify the composition desired.

In order to obtain a better idea of the present practice and trend in the testing of rubber insulation on cables and the relative value and the significance which can be given to the various tests, an inquiry was sent to a number of producers and consumers of rubber-insulated cables, the Underwriters' Laboratories, the National Bureau of Standards,[2] the United States Coast Guard and the U. S. Bureau of Mines as to their practice.

A considerable proportion of them cooperated by furnishing a statement of their practice in testing and their comments on the various tests. Some consumers reported that they relied upon manufacturer's guarantees and tests made by the manufacturer.

While the survey does not include a very large number of companies on account of the limited time available, replies were received from companies representing various industries and using considerable quantities of cable in widely different applications. The cable manufacturers readily cooperated in the work.

The present practice in the testing of rubber insulation for the various classes of service as reported, is shown in Table I.

A summary of the comments on the principal tests is given below:

Chemical Analysis

Underwriters' Laboratories:

Building wires and flexible cords are practically the only types of rubber-insulated conductors listed and inspected by the Laboratories. Our listing of rubber-covered building wires covers only "code" wire. Our listing of flexible cords covers "code" rubber, 30 per cent rubber and 40 per cent rubber insulation. The chemical tests applied have been used by us

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[1] Materials Engineer, Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] The material used has been summarized from letters of March 23 and April 11 from Dr. A. T. McPherson, Chief of Rubber Section.

TABLE I.—TESTS WHICH ARE BEING MADE ON RUBBER INSULATION OF CABLES.

	Cable Manufacturers							Underwriters' Laboratories			Federal Specifications					United States Coast Guard	U. S. Bureau of Mines	Railroad No. 1			Public Utility No. 1	Public Utility No. 2			Public Utility No. 3	Public Utility No. 4	
	Building Wire	Underground and Submarine Power Cables	Corona Resistant	Heat Resistant	Rubber Jacketed	Submarine Telephone	Telephone and Control	Code Building Wire	Code Flexible Cord	30 and 40 per cent Rubber Flexible Cord	Insulated Cables, Proposed 1:8:36	Rubber Armor for Cables, Proposed 1:8:36	Code Building Wire J-C-101a	Superaging Building Wire J-C-106 (0–5000 v.)	Superaging Wire (Not Building) J-C-121 (0–8000 v.)	Telephone and Telegraph	Mine Car Cables	30 per cent Rubber	Code Rubber	60 per cent Rubber Jacket	Rubber Insulation General Purpose	Class AO	Class NW	Class HV	Rubber Insulation All Applications	Underground	Overhead
Chemical analysis	√							√	√	√			Rubber	√	√			√		√		√					
Tensile strength	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√		√	√	√	√	√	√	√	√	√	√
Elongation	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√		√	√	√	√	√	√	√	√	√	√
Set	√	√	√	√	√	√	√	√	√	√								√	√	√	√	√	√	√	√	√	√
Modulus of elasticity	√	√	√	√	√	√	√					√				√											
Specific gravity																		√				√					
Compression cutting					√	√	√					√				√											
Abrasion resistance					√																						√
Tear Resistance					√							√				√											
Compression (7 ton car)																	√										
Geer oven, 70 C	√	√	√		√	√	√	√					√			√					√	√	√	√		√	
Geer oven, 120 C				√																							
Oxygen bomb, 70 C., 96 hr	√	√	√		√	√	√	√		√		√	√			√					√	√	√	√	√	√	
Oxygen bomb, 70 C., 240 hr																					√						
Air bomb, 260 F., 80 lb				√							√			√	√												
Light checking			√		√																				√		√
Ozone			√																							√[a]	
Corona			√																					√		√[a]	
Water absorption		√	√	√	√	√	√					√				√					√	√	√			√	
Conductor corrosion		√	√	√		√										√											
Absorption, oil, etc																							√				
Dielectric constant and power factor		√	√	√		√										√											
Change in specific inductance capacity and power factor in immersion in water		√	√	√		√										√										[a]	
Dielectric strength		√	√	√		√		√			√		√	√	√	√		√	√		√	√		√	√	√	[b]
Insulation resistance	√	√	√	√		√	√	√			√		√	√	√	√		√	√		√	√			√	√	
Electrostatic capacity																√											
Cold bending																										√	

for some 30 yr. with relatively little change. They constitute a well-established method of judging the quality and performance of rubber compound, rather than a test to determine the life or performance in actual use as insulation.

Bureau of Standards:[2]

There is a general tendency to discard all chemical analyses in the examination of rubber. Modern developments in rubber compounding have rendered many specifications based on composition not only obsolete, but even barriers to the customers best interests. In my opinion there is, however, one composition requirement worth keeping, namely the rubber content. The physical, mechanical and electrical properties of rubber compounds all have an intimate dependence on the rubber content so that in order to get certain desired characteristics it may be simpler and more convenient to specify the rubber content than to specify a group of physical properties.

The rubber content on which the various properties of a compound depend is a function of the percentage of rubber by volume. Rubber content by weight is without practical significance, unless the specific gravity is stated also. One rubber compound containing 40 per cent of rubber by weight may have in it less rubber by volume than another compound containing 30 per cent by weight. Unless there is some special reason for fixing the specific gravity of the rubber insulation, the only fair means of specifying rubber is on a volume basis.

United States Coast Guard:

It has been our policy in recent years to judge all rubber compounds by the various performance tests outlined, and we feel that this assures the desired properties better than chemical analysis of the compounds.

Bureau of Mines Experiment Station:

Trailing cables for mine cars are given a performance test as indicated under "Mechanical Tests."

Consensus of Cable Manufacturers:

As a result of years of experience, many consumers of wire and cable have come to rely on chemical analysis to identify satisfactory types of insulation. Some insulations bought to chemical tests have good service records. By only slight modifications in the limits of certain chemical requirements, it is possible to make use of this service record and still utilize improvements in the science of rubber compounding. To this extent chemical tests serve a very useful purpose. Generally, however, performance tests without restriction on the type of compounding ingredients used are a more useful criterion of satisfactory service where past experience is not available or new requirements are to be met, and in these cases chemical requirements have been found to be a detriment. Most of the information given by chemical tests can be obtained by performance tests which are simpler; and because they represent a larger total of the actual production they are more reliable.

Cable Manufacturers Nos. 1 and 2.—We agree that chemical tests are useful when used to identify insulations which have proven satisfactory in service. Generally, however, we believe that chemical tests have little or no practical value. This is true for the simple dry mineral compounds which are easily analyzed, and is especially true for special stocks containing a number of organic materials. We can see no value in specifying rubber content if no limitation is placed on the other ingredients in the rubber compound. A requirement for rubber by volume is too indefinite to be of any practical value. If restriction is made in compounding so that rubber by volume may be accurately determined, the old form of chemical analysis is specific and definite, while the general requirement without such analysis is meaningless.

Cable Manufacturer No. 3.—While generally agreeing with the statement of the cable companies, I know of no other test which will give the percentage of rubber. If performance specifications could be written with all of the performance tests needed to define exactly all of the characteristics of a rubber compound, it would be useless to use any chemical test to determine the percentage of rubber. However, in the absence of complete performance tests a purchaser may wish to specify the quality of the insulation by calling for a compound of a definite percentage of rubber.

Cable Manufacturer No. 4.—We agree with the statement of the cable companies. Specifying the minimum rubber content is helpful, especially in view of the general acceptance of rubber content as a means of designating the quality of rubber insulation.

Railroad No. 1:

We believe that certain chemical determinations are required in coordination with aging and tension tests as a check, in order to insure proper life, but we are not in a position at present to give these definitely.

Public Utility No. 1:

We do not particularly care for chemical tests for rubber compounds much preferring electrical, physical and life tests of various sorts, in an attempt to demonstrate the ability of the rubber to withstand service conditions.

Public Utility No. 2:

Class AO rubber compound (with chemical analysis) is used for overhead primary and secondary lines, metallic sheathed underground series lighting and station control wiring. Class NW used for all underground secondary mains and services, and class HV for primary distribution pole lateral cables are purchased on performance tests without reference to composition.

Public Utility No. 3:

The manufacturer should not be hampered by composition specification, but should be given the opportunity to develop material combinations resulting in a superior product. The criterion of performance must lie in service records, or where possible, and in most cases it is possible, on tests devised to approximate the service conditions. Accelerating features must necessarily be introduced. The so-called accelerated aging tests though often open to question are far more capable of evaluating a product than the mere specification of materials which may be compounded under numerous and even unknown conditions.

Public Utility No. 4:

The use of chemical analysis to control the quality of rubber insulation is, in our opinion, entirely the concern of the manufacturer. From his standpoint it is exceedingly important for variations of insulation characteristics are primarily a matter of the composition of the finished product. The user of cable, however, is interested solely in obtaining a product which will meet certain physical requirements and have as long a life as possible. How this is accomplished is not particularly his concern.

MECHANICAL TESTS—TENSILE STRENGTH, ELONGATION AND SET

Underwriters' Laboratories:

The tensile strength, recovery and stretch tests provide a check upon the rubber compound and its vulcanization.

Bureau of Standards:

The usual mechanical tests are tensile strength and ultimate elongation, neither of which bears any direct relation to the performance of the insulation. They are employed because they are traditionally used for rubber and because they afford an indirect measure of other properties which are related to serviceability, but which cannot be so readily measured.

An effort is being made in various quarters to correct this situation by the development of service-related tests, such as compression cutting, tear resistance, etc.

The need for tests other than tensile strength measurements has been emphasized by the recent increase in the use of very thin-walled rubber insulation. It is often impossible to make reliable tensile strength measurements on the very thin rubber, but the very fact that the rubber is thin renders its mechanical properties all the more important.

United States Coast Guard:

Tests for tensile strength, elongation and modulus of elasticity are made to insure the desired physical properties.

Cable Manufacturers:

Tensile strength, elongation, modulus of elasticity and permanent set determine the general type of compound, its approximate rubber content, and from lot to lot, are a measure of uniformity in the compound. Physical tests also are the measuring stick for accelerated aging tests.

Railroad No. 1:

The tensile strength and elongation undoubtedly serve as a measure of the relative aging characteristics. Certain strength and elongation values should be obtained for constructional reasons, varying possibly for the various uses.

Public Utility No. 4:

Tensile strength is a measure of the "liveliness" or freshness of the rubber.

AGING TESTS

Underwriters' Laboratories:

The only test which can be considered as a basis for evaluating the life of rubber compounds is the oxygen bomb test. The requirements for this test were included in the Underwriters' Laboratories Standard for Rubber Covered Wires and Cables in August, 1935, and are being studied in connection with the long-established chemical tests for one year. Then we will endeavor to determine whether chemical tests can be abandoned in favor of the oxygen-bomb test.

Bureau of Standards:

On account of the diversity of service conditions and shortcomings of the test methods, it is not yet possible to employ them for making any accurate forecast in years of the service life of any given cable. This does not mean that aging tests are without value. On the contrary, they are of considerable importance even though precise quantitative significance cannot be attached to them. On account of this uncertainty, I am of the opinion that the use of two or three different tests with moderate limits is a better means of securing a durable product than a single test with a very drastic requirement.

In view of the extensive use of rubber insulated cable in water, underground, or in moist locations, I am of the opinion that more emphasis should be placed on the effect of water and that new tests are needed not simply to measure water absorption but rather to produce accelerated aging in the presence of water.

Tensile strength measurements on aged samples are significant in so far as they serve to measure depreciation, hence it is the percentage change in tensile strength after aging, not the actual tensile strength which is significant.

United States Coast Guard:

We are using both the oxygen-bomb test and the Geer oven test to determine aging.

Cable Manufacturers:

The 70 C. Geer oven and the oxygen-bomb tests determine normal aging characteristics. They do not indicate resistance to cracking when exposed to light, or weather aging. When run in comparison with a compound of known performance, they give a reliable index of expected life.

The 120 C. Geer oven and 127 C. air bomb are extremely severe tests useful only in determining heat resistance. They are too severe to use on normal compounds and are limited to heat-resistant compounds. These latter stocks. however. will also show excellent normal aging.

Railroad No. 1:

Aging tests alone do not give definite assurance of long service life. We feel that certain chemical determinations are required in coordination with aging tests to insure proper life.

Public Utility No. 1:

We are using the oxygen-bomb test and the Geer oven test. We believe that some of the standards we use do not carry these tests for a long enough time—for instance the 96-hr. oxygen-bomb test, to demonstrate that the compound actually will have a long life. The shape of the depreciation curve should be of great value. In addition, certain minimum physical requirements after exposure to accelerated life tests may be of greater interest than the percentage depreciation from initial physical values.

Public Utility No. 2:

The oxygen-bomb test and Geer oven test are used.

Public Utility No. 3:

The oxygen-bomb test is used at 70 C. for 250 hr., and the tensile strength, elongation and dielectric strength are determined after aging. Work is being done to evaluate bomb aging with life in service.

Public Utility No. 4:

Accelerated aging is a measure of the resistance of the rubber to heat and oxidation.

CRACKING DUE TO LIGHT AND WEATHER AGING

Cable Manufacturers:

A test of this type is applicable to rubber compound exposed to sunlight or weather or both. It is generally used on rubber-jacketed cords which have no outer protective covering. There is no standard method of making this test.

Public Utility No. 4:

Weatherometer test is an accelerated test designed to show the effects of various kinds of weather upon the life of the insulation.

COMPRESSION CUTTING

United States Coast Guard:

This test is made upon the original samples and on those aged in the oxygen bomb, and aged in water at 70 C. It is more rapid than the tension tests for control purposes where many tests are required.

Bureau of Mines Experiment Station:

Performance tests of trailing cables are made in which the cable is stretched across track rails and a 7-ton car is run over it. This test is for the purpose of proving the toughness and strength of the sheath and the resistance of the conductor insulation to crushing. The cable must not short circuit nor ground to rails or wheels at more than 10 per cent of the 100 places where two wheels have passed over it.

Cable Manufacturers:

This test was developed specifically for telephone drop wire, and is useful on compounds subjected to crushing loads. It offers a measure of hardness and toughness, but has not yet been correlated with other physical tests.

It has been used for 20 yr. on telephone wire, offering a test of direct application as compared with the tension test which has value only by indirection.

ABRASION RESISTANCE

Cable Manufacturers:

This test is a measure of wear resistance. It is a fundamental test in development of tough rubber jacket compounds. The test is made on laboratory-prepared samples as it is difficult to secure suitable specimens from cable jackets.

Public Utility No. 4:

This test is intended to simulate as nearly as possible the rubbing of a wire against a tree.

TEAR RESISTANCE

Cable Manufacturers:

Another test used on jacket compounds. It is closely related to abrasion resistance and may be determined when abrasion tests are difficult to perform.

United States Coast Guard:

The tear test is made on jacket stock to insure a tough, high-carbon stock.

COLD-BEND TEST

Public Utility No. 4:

As a test for brittleness when exposed to low temperature, short samples of underground cable with all braids removed are brought to uniform temperature at –17.7 C. (0 F.) then bent around a mandrel five times the diameter of the cable.

OZONE - CORONA

Cable Manufacturers:

Rubber, particularly when put under a slight tension, deteriorates rapidly when subjected to ozone. This test gives valuable information on compounds which encounter ozone or corona formation in service. The test has little value for regular stocks, but offers an excellent test for ozone-resistant compounds. It is generally used for high-voltage cables.

Public Utility No. 2:

A corona test is used on high-voltage insulation.

Public Utility No. 3:

High-voltage cable insulation is subjected to dry ozone, while under tension. It is also subjected to a test for corona formation.

Public Utility No. 4:

The ozone-resistance test in which a bent sample is exposed to ozone determines the resistance of the exposed surface of the insulation to the action of ozone.

The test for corona point for so-called corona-proof cables, made on bent sample with increasing voltage, determines the voltage at which corona first appears.

The grounded plate test in which a bent sample is tested is a mild form of ozone test with the cable subjected to moderate over-voltage.

WATER ABSORPTION

Consensus of Cable Manufacturers:

The measurement of water absorption is a relatively new test for insulated wire, although submarine cables have been made for many years. A considerable proportion of wires and cables in service is subjected to moist conditions, and a water-absorption test is of prime importance for such installations.

Both a mechanical water-absorption test (the increase in weight during immersion) and an electrical method of measurement by change in specific inductance capacity are used. Neither test at present is considered entirely satisfactory. Work is under way to develop a satisfactory test. In places where considerable moisture exists, the life of the rubber insulation is measured by its resistance to moisture rather than by its physical and mechanical properties. When the insulation is short circuited by moisture it is no longer a dielectric.

Some cable manufacturers agree to the above statement and state that a mechanical water-absorption test will give considerable information, although for severe conditions it should be checked with electrical measurements.

Other cable manufacturers believe that a mechanical water-absorption test does not yield any useful information as to how rubber-insulated cable will act under wet service conditions.

United States Coast Guard:

Mechanical water-absorption test is made to insure compounds that do not absorb excessive quantities of moisture. Electrical water-absorption test (capacity between coil and ground, after 1, 7 and 28 days immersion in water at 70 C., 1000 cycles per second) is made to insure compounds that do not show undue impairment of electrical properties when immersed in water. Both mechanical and electrical tests are specified to reduce the possibility of "trick compounds" designed to merely meet certain specified limits.

Public Utility No. 1:

We are very much interested in seeing an adequate test formulated to determine the moisture-absorption characteristic of rubber insulation. At the present time there are three tests being made:

1. The change in specific inductance capacity as outlined in the A.S.T.M. specifications[3] for performance and class AO rubber compounds.

2. An absorption test in terms of weight of water absorbed by the rubber sample after seven days immersion, in percentage of the total weight of the sample.

3. The absorption of water in terms of weight absorbed by the rubber samples in percentage of surface area of the sample.

We do not particularly like the specific inductance capacity method, much preferring one of the two types of weight determination, but in these cases we think it necessary that some agreement be reached as to the shape and thickness of the sample tested. This is especially true where the weight of water absorbed is expressed as a percentage of the total weight, since thin strips having large surface areas in proprotion to the total weight may show up more poorly on the basis of percentage of weight than in terms of weight even though the compound be otherwise the same.

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[3] Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 35 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1115 (1935); also 1935 Book of A.S.T.M. Tentative Standards, p. 1135.

Tentative Specifications for Insulated Wire and Cable: Performance Rubber Compound (D 353 - 35 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, 1141 (1935); also 1935 Book of A. S. T. M. Tentative Standards, p. 1161.

Public Utility No. 4:

The test determines the resistance of the cable to the absorption of water. Specimens about $2\frac{1}{2}$ by $\frac{3}{8}$ by $\frac{3}{64}$ in. are immersed in distilled water at 70 C.

CONDUCTOR CORROSION

Cable Manufacturers:

In connection with moisture resistance the tendency toward conductor corrosion should be observed. A compound having poor moisture resistance together with a tendency to corrode the tinned copper conductor will not stand up long in service. Corrosion taking place at the conductor and moisture penetrating the insulation will rapidly produce a cable failure. Corrosion tests are of much value in submarine cables and other wires subjected to severe moisture conditions.

United States Coast Guard:

A copper-corrosion test is made to insure insulating compounds that will not react with the copper in the presence of moisture and cause excessive corrosion.

ELECTRICAL TESTS

Bureau of Standards:

The dielectric constant of the rubber insulation is important for certain communication cables, the power factor and the dielectric strength are important for power cables operating at high voltages, but for the great bulk of electrical insulation, the electrical properties of virtually any well-made rubber compound are fully adequate to meet the actual electrical requirements. In such cases, the principal function of the electrical tests is not to measure any significant electrical property but to determine whether the rubber insulation is continuous and free from mechanical faults.

Building wire, for example, would probably serve its purpose equally well with only one hundredth the insulation resistance commonly specified provided the rubber sheath were free from flaws and incipient defects. The present-day insulation resistance requirements are based on the classical, dry-mineral filler compound without organic accelerators or antioxidants, and may be unfair when applied to modern compounds having inherently different basic properties.

Another instance in which sight of the purpose of the test is sometimes lost occurs when an insulation-resistance test is made on short lengths of wire. Such tests have no practical significance because the purchaser is not concerned with the electrical properties of the rubber but with its freedom from flaws and defects, which can be ascertained only by testing a considerable length.

Cable Manufacturers:

Electrical properties are the primary considerations for all insulating compounds. Emphasis varies depending upon the type of service for which the cable is being used. Building wire used at 600 v. and lower should be uniform and continuous, but does not demand exceptionally high electrical properties. Jacket compounds need no electrical properties. A submarine telephone compound should have a low dielectric constant and a low power factor, but a high dielectric strength is not necessary.

Dielectric constant, power factor and dielectric strength are most valuable in determining the suitability of a rubber compound. Insulation resistance is easily measured but high values offer no guarantee as to the quality of the insulation. The test is subject to climatic conditions and varies from day to day.

Railroad No. 1:

Undoubtedly the electrical tests are important. Insulation resistance has been used effectively in checking over various experimental installations.

Public Utility No. 3:

Dielectric strength, which for years was ignored, is fully as important for consideration as the mechanical properties, particularly after oxygen bomb aging. Insulation-resistance tests also are made.

Public Utility No. 4:

Factory high-voltage test on entire cable determines ability of cable to withstand prolonged over-voltage. Twelve hours immersion in water, then 15-min. test.

Double voltage test on 6-ft. samples determines ability of cable to withstand severe over-voltage for short periods. Twelve hours immersion in water, then 5-min. test.

Breakdown test after double voltage test determines the ultimate electrical strength of the cable on momentary over-voltage.

The intermittent voltage test on overhead wires simulates the action of a live wire swinging to and from a grounded object.

The insulation resistance test follows the factory high voltage test, and determines the resistance to the passage of current.

Power factor gives a measure of dielectric loss. It is made on used cables.

GENERAL COMMENTS

Cable manufacturers state that they believe that several carefully chosen tests which closely simulate service conditions will offer a good measuring stick for predicting the life of a rubber compound under ordinary conditions. However, they cannot take into account accidental conditions which may arise and injure the cable. Performance tests should be designed particularly for the service for which the cable is intended, and not considered as applying to the rubber alone.

The comments which have been quoted show the pronounced trend away from chemical requirements toward performance tests, and the need for improvement and extension of performance tests to provide better control of quality. There is also need for further work in interpreting aging tests and correlating them with life in service. Requests were made for more adequate tests for moisture absorption and resistance to sunlight and ozone. Improvements in other tests for various applications also were suggested.

Cooperation between producers and consumers in developing more effective tests and interpreting them in terms of service is necessary to secure the best results.

Acknowledgment.—I wish to express my appreciation of the fine spirit of cooperation which has been shown by many companies, both producers and consumers, and Government departments in preparing this survey.

DISCUSSION

Mr. R. W. Chadbourn.[1]—From the variety of opinions presented on the question of rubber cable testing, it appears that present test standards are not universally acceptable and that it would be very desirable if we could devise simple and inexpensive service tests which would give us some idea of the condition at the moment and the probable future life of the cable. The problem is not an easy one. The cable engineer is at a distinct disadvantage in comparison with the user of certain other rubber products. It is possible, for instance, to put a tire on an automobile and get some idea as to the expected performance of that tire under service conditions in a fairly short time.

The cable engineer cannot do that; when he buys and installs a piece of cable he expects the cable, if used in accordance with standard practice, to last forever. Unfortunately, rubber as an insulating material is a variable product, and in the forms most familiar to engineers is subject to more or less deterioration, which depends upon many factors. The deterioration of cable may be anticipated by the use of some sort of an accelerated test, and various accelerated tests have been devised for that purpose. Unfortunately, no accelerated test has ever been invented which duplicates service use. The opinions expressed in Mr. Harvey's paper reflect the existing disagreement on the importance of accelerated tests. How far these can be improved it is impossible to say.

The question therefore arises as to what can be done in the way of service tests. So far not much has been accomplished, but I should like to suggest one or two possibilities. The chief difficulty, of course, is the time factor. It is obviously undesirable to be obliged to wait many years in order to find out how good a cable really is. During that period the art of manufacture may have progressed so far that the cable in question may have become obsolete, in which case the test data would cease to be of value.

In my own company, however, it has been possible to obtain some real information on sections of cable which for some reason or other have been removed from service. The amount of cable so removed, while small compared to the total amount of cable used, is sufficient to give us considerable data over a period of a few years. The fact that this cable can be tested in the laboratory under controlled conditions makes such data of particular value. We have found, for instance, that power factor is an excellent criterion of cable condition—the power factor increasing at a rapidly increasing rate as deterioration progresses.

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[1] Assistant Superintendent, Standardizing and Testing Dept., The Edison Electric Illuminating Company of Boston, Boston, Mass.

It is not possible to do quite so much on cable in service for various reasons, chief of which is that the cable temperature is not easily controlled. In most installed cables the temperature is variable from one end to the other. As the electrical properties of the insulation vary widely with temperature and as it is difficult on such cables to obtain a fair average temperature, it is obvious that tests made on such lines are subject to considerable error. On most systems, however, there are a few lines operating at approximately uniform temperature. Periodic power factor tests on such lines, coupled with accurate determinations of average temperature, may give us some valuable information.

I mention these items at this time to show the possibilities of correlating service tests with laboratory tests, the latter being the sole criterion which most of us now have for judging cable condition.

MR. R. A. SCHATZEL.[2]—The comments summarized in the paper illustrate that the judging of insulated wire is quite different from the judging of other rubber products. In other uses, rubber is not considered as rubber alone, but as a part of an automobile; as a part of a hose; or as a part of a tire; and the particular function it was going to perform in that particular place is considered, and, therefore, the test is designed to determine its suitability.

As far as wire and cable are concerned, rubber to us is not rubber; it is a dielectric, and the first function of any test to show whether it will operate satisfactorily is whether it will operate as a suitable dielectric for the particular purpose it is supposed to perform. Then we may apply to it the standard tests that are used in testing rubber and gage its uniformity, state of cure and other properties which will show us that from lot to lot we are getting the same dielectric. To some extent that has been overlooked and we often judge rubber to be a good dielectric because it has a high tensile strength. The fact that it has a high tensile strength has absolutely no relation to its dielectric properties. As a matter of fact, two rubber compounds having the same aging properties by no means can be considered to be of the same dielectric.

Then there is another tendency in most of these performance tests. We are making performance tests which are really tests for uniformity and are trying to put them into a specification so as to make them convenient to perform. The objective is first to find out what are good tests, and the suggestion has been made by Mr. McPherson that two or three or more moderate tests to bring out various properties furnish a better criterion than one single severe test. At present, however, we are making tests severe, considering their very severity a criterion of their good quality. I think the survey which Mr. Harvey has so well made will serve a very useful purpose if it does nothing else than to point out that that is not the direction in which to go, and that perhaps we must first find out what are good tests for rubber as a dielectric, and then superimpose on them some convenient tests which will check the uniformity.

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[2] Director of Research, General Research Laboratories, General Cable Corp., Rome, N. Y.

MR. A. H. NUCKOLLS[3] (*by letter*).—I regard this paper as an important contribution to the literature on the subject. It gives briefly a survey of the present practices and trends in the testing of rubber insulation, and brings out important differences in the viewpoints of many producers and consumers on the principal tests in use.

The Underwriters' Laboratories, while using chemical and physical tests for about 30 years, the results of which compare favorably with results of natural aging tests, favor performance tests in lieu of composition tests, and are at the present time conducting oven and oxygen bomb aging tests. Experimental work is also being done on various other methods of test, including accelerated light tests. As stated in the paper, requirements for the oxygen bomb test as a basis for evaluating the life of rubber compounds for insulation are being used and studied at Underwriter's Laboratories in an endeavor to determine whether long-established chemical tests can be abandoned.

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[3] Chemical Engineer, Underwriters' Laboratories, Chicago, Ill.

THE RATE OF REACTION OF SODIUM SULFITE WITH OXYGEN DISSOLVED IN WATER

By R. M. Hitchens[1] and R. W. Towne[1]

Synopsis

Sodium sulfite is being employed in increasing amounts for the prevention of corrosion of metal surfaces by dissolved oxygen. Since its effectiveness is dependent upon the speed with which it combines with dissolved oxygen, information on this subject, concerning which the literature is fragmentary, is needed.

It was the purpose of this investigation to study the effect of the temperature, of the type of water, and of the concentration of sodium sulfite, upon the reaction rate.

The reaction proceeds about five times as rapidly in ocean water as in distilled water and ten times as rapidly in distilled water as in St. Louis, Mo., city water. It proceeds with great rapidity at 85 C. even in the last-named water, which possesses strong inhibitory properties, although the reaction is slow in this water at low temperatures. A 20 per cent excess of sodium sulfite almost doubles the reaction rate and a 100 per cent excess almost quadruples it.

From the data obtained it seems logical to conclude that the reaction proceeds with great rapidity and that it is complete in less than 1 min. at temperatures approaching that of boiling water, regardless of the type of water, if a slight excess of sodium sulfite is employed.

Introduction

Corrosion of iron due to oxygen dissolved in water has long been a serious problem. Efforts to remove dissolved oxygen, especially in steam boilers, have been directed along two lines—the mechanical method and the chemical method. Mechanical deaeration, the most widely used method, is the most economical for the removal of the bulk of the oxygen. At the present time the mechanical method is often supplemented by chemical deactivation to remove the last traces of oxygen. A summary of the methods used with a discussion of their application is given by Powell.[2]

Sodium sulfite, which combines readily with oxygen to form sodium sulfate, is now employed extensively to remove oxygen from water. This is not not a new process, having been patented in several countries in 1913 by Gans. However, it is only within the last four years that its commercial application has become extensive.

Sodium sulfite offers some advantages not possessed by other chemicals used for removing oxygen from water. It is economical, easy to apply and easy to control by simple analytical tests. It is soluble in water and is oxidized to water-soluble sodium sulfate, the presence of which in boiler water has been considered an asset. A discussion of the utility of this method of chemical removal of oxygen together with general instructions for its application is given by Powell.[2]

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[1] Assistant Chief Analyst, and Chief Analyst, respectively, Analytical Laboratories, Monsanto Chemical Co., St. Louis, Mo.

[2] S. T. Powell, "Chemical Deaeration of Boiler Feed Water," *Power* Vol. 79, pp. 304–306, 362–363 (1935).

Fears that sodium sulfite would hydrolyze at boiler temperatures to form sulfur dioxide which might be carried over with the steam have proved groundless. The present authors have demonstrated the complete absence of such carry-over of sulfur dioxide at low pressures. A central station company[3] has proved the absence of sulfur dioxide in the steam from boiler water containing 100 p.p.m. of sodium sulfite at temperatures corresponding to 1200 lb. per sq. in. pressure, in experimental equipment.

The chief questions regarding the use of sodium sulfite have pertained to its rate of reaction with dissolved oxygen and to the completeness of the reaction. Marguerre,[4] describing in detail the troubles encountered with a 1400 lb. per sq. in. pressure boiler at Mannheim, Germany, states that oxygen corrosion did not cease until sodium sulfite treatment was adopted. Such treatment, he says, reduced the oxygen content of the boiler water to less than 0.01 p.p.m. Recently, Purdy,[5] investigating the reaction between sodium sulfite and oxygen in feed water to a marine boiler, accounted for complete removal of oxygen in 1 min. both from the increase in sulfate and from the decrease in sulfite content of the water. In addition, a modified Winkler test showed zero oxygen in the water at the heater exit.

The present investigation was undertaken for the purpose of obtaining under a variety of conditions laboratory data concerning the rate of reaction of sodium sulfite with oxygen dissolved in water.

The reaction between pure sodium sulfite, dissolved in pure water is immeasurably slow, if it occurs at all. The reaction does not proceed at a measurable rate unless a catalyst is present. The catalysts for this reaction are the cations of the heavy metals which show more than one valence. The cupric ion is the most powerful, exhibiting marked catalytic properties when present in a concentration as low as 10^{-12} mols per liter. Other marked catalysts are the ions of iron, cobalt, nickel and manganese. Anions apparently exert no catalytic effect; neither do cations of such metals as the alkalies or the alkaline earths.

Assuming that sufficient catalyst is present to allow the reaction to proceed, the rate of reaction is a function of the hydrogen-ion concentration of the reaction medium. Bigelow,[6] Titoff,[7] and Reinders and Vles[8] each found that the maximum rate of reaction occurs at pH values from 9 to 10, the pH range of aqueous solutions of sodium sulfite. This indicates that the sulfite ion is the particular portion of the molecule which is oxidized. All of these investigators found that as the solution is made more acid the rate of reaction diminishes.

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[3] Private communication.

[4] Marguerre, "Experiences with High-Pressure Boiler Plants," *Engineering*, Vol. 132, pp. 267–271 (1931).

[5] A. C. Purdy, "Investigation of Sodium Sulphite for Removing Oxygen from Boiler Feed Water," Private communication.

[6] S. L. Bigelow, "Katalytische Wirkungen auf die geschweidigkeit der oxydation des natriumsulfits durch den Sauerstoff der Luft," *Zeitschrift für physikalische Chemie*, Vol. 26, p. 495 (1898).

[7] A. Titoff, "Beiträge zur Kenntnis der negativenkatalyse im homogenen System," *Zeitschrift für physikalische Chemie*, Vol. 45, p. 641 (1903).

[8] W. Reinders and S. I. Vles, "The Catalytic Oxidation of Sulphites," *Recueil* des travaux chimiques des Pays-Bas, Vol. 44, pp. 249–268 (1925).

They found also that as the pH value of the solution becomes higher than 9 to 10 the reaction rate diminishes. Since the sulfite ion concentration does not diminish as the solution is made alkaline they attribute this to the reduction in the concentration of the catalytic ions through their precipitation or their transformation into complex ions.

Substances which form complexes with the catalytic ions retard the reaction. They include most organic and some inorganic compounds. They sometimes have been called negative catalysts. In reality this is a misnomer since such substances affect not the rate of the reaction but rather the concentration of the catalyst.

The rate of reaction becomes more rapid with increase in temperature, in general, an increase in temperature of 10 deg. Cent. doubling the speed of reaction.

Although considerable work has been carried out on the rate of the reaction, practically none of it can be utilized to predict how rapidly and how completely sodium sulfite will remove oxygen from water. Kobe and Gooding[9] recently completed an investigation with this end in view.

Their method consisted of adding a known quantity of sodium sulfite to a sample of water of known oxygen content, stopping the reaction at a specified time with an excess of iodine and titrating this excess with sodium thiosulfate. Thus the oxygen removed is calculated from the loss in sodium sulfite and is not observed directly. It would be better to measure the oxygen remaining after the reaction is stopped since this would give more accurate data concerning its completeness. Unfortunately there is no method for the direct determination of dissolved oxygen which allows the freedom and speed of handling necessary to carry out rate of reaction experiments. Thus the indirect method of Kobe and Gooding,[9] with some modification in technique, is the one used in obtaining the data in this paper.

EXPERIMENTAL WORK

In this work it was planned to study the rate of reaction of sodium sulfite with dissolved oxygen at 15-deg. intervals from 10 to 85 C. These experiments were to be carried out with the addition of the theoretical quantity of sodium sulfite to remove all of the oxygen and also with the addition of both a 20 per cent and a 100 per cent excess of this reagent. Since there is ample evidence that the reaction rate is affected by the type of water used, it was decided to carry out experiments in water from different sources. Distilled water was chosen as a type containing but minute traces of impurities, both catalytic and inhibitory. St. Louis, Mo., city water was chosen as a second type after examination of several natural and boiler waters had shown it to exhibit a marked retarding effect upon the reaction. Atlantic Ocean water, collected several miles out from Boston, Mass. and shipped in glass containers, was chosen as another type since previous experiments had shown it to exercise a marked catalytic effect.

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[9] K. A. Kobe and W. L. Gooding, "Oxygen Removal from Boiler Feed Water by Sodium Sulphite," *Industrial and Engineering Chemistry*, Vol. 27, pp. 331–333; 1935).

Since expense precludes the practicability of using chemically pure sodium sulfite for removing oxygen from water and since small amounts of metals such as would be present in the technical material enhance the reaction rate somewhat, it was decided to use a technical grade of sodium sulfite throughout this investigation. Comparison of the results obtained using the same sample of water under identical conditions showed that this technical product reacted with dissolved oxygen about ten per cent more rapidly than did chemically pure sodium sulfite.

Materials:

Sodium sulfite, a technical anhydrous grade containing approximately 95 per cent sodium sulfite, 0.1 per cent insoluble matter, the remainder being sodium sulfate. The material used consisted of a representative sample of a technical variety.

Standard Solutions:

Iodine 0.1 *N*, standardized against arsenious trioxide.

Sodium sulfite, 0.1 *N*, standardized against the 0.1 *N* iodine. This solution was renewed each day.

Sodium thiosulfate, 0.01 *N*, standardized against potassium iodate.

Soluble starch, 1 per cent solution.

Test Method:

The following procedure was found to give reliable results:

The water under question was adjusted to the temperature desired by immersion in a thermostat. The water was then saturated with air by passing a stream of air through it for at least 1 hr., the oxygen content of this water being determined by the Winkler[10] method. The barometric pressure under which the aeration was carried out was noted. From the change in pressure from day to day, any slight deviation in the oxygen content of the water was calculated. This method of obtaining water of known oxygen content was found to be reliable.

A 250-ml. Soxhlet extraction flask was filled with this water, the weight of water adjusted quickly to 250 g. and the flask reimmersed in the thermostat. It was closed immediately with a rubber stopper fitted with an inlet tube ending just above the water level, an outlet tube closed with a bunsen valve and a hole into which a small rubber stopper bearing the tip of the burette holding the sodium sulfite solution was inserted. The air above the water was replaced with nitrogen by introducing a slow stream of this gas for about 1 min. The calculated volume of sodium sulfite solution was added. The reagent was mixed quickly and thoroughly with the water by swirling the flask. The time for the start of the reaction was taken as the instant at which the first drop of sodium sulfite solution touched the water. The reaction mixture was maintained at the desired temperature throughout the reaction. Immediately prior to the end of the reaction time the stopper was removed from the flask. At the exact expiration of the reaction period 2 ml. of glacial acetic acid and 1 g. of potassium iodide were added. Without delay the reaction was stopped completely by adding an excess of standard iodine solution. The temperature of the water was adjusted to 25 C. and the excess iodine determined by titration with standard sodium thiosulfate solution, using 1 ml. of soluble starch solution as indicator. A sketch of the reaction flask used is given in Fig. 1.

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[10] H. S. Lewis, "Measure Feed Water Oxygen this Trouble Saving Way," *Power*, Vol. 78, pp. 510–512 (1934).

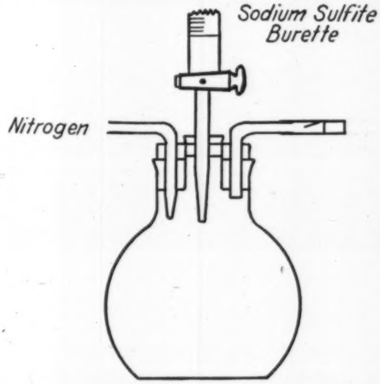


FIG. 1.—Reaction Flask.

It was found necessary to remove oxygen from the gas phase to prevent its absorption by the water and consequent reaction with sodium sulfite, otherwise errors from this source often amounted to 50 per cent.

Addition of acetic acid and potassium iodide was required to stop the formation of iodate upon the addition of excess iodine. This reaction occurred extensively at the higher temperatures. Acetic acid alone did not prevent it.

The pH values of the water samples containing sodium sulfite were determined potentiometrically by means of the glass electrode.

Test Data and Discussion

The results obtained by this procedure using distilled water, are shown graphically in Fig. 2. Tests were run at 10, 25, 40, 55, 70 and 85 C., but data are included for only those temperatures where the results are measurable.

Fig. 2 (*a*) contains the data obtained when sodium sulfite was added in the exact amount required to react with all the oxygen present in the water. It will be seen that the reaction is only moderately rapid at 10 C. but that it is accelerated rapidly by increasing temperatures until, at 55 C., oxygen removal is practically complete in 1 min. and at 70 C. is complete in 30 sec. At 85 C. it is too rapid to measure. It is undoubtedly complete in much less than 30 sec., which time interval is the shortest in which the reaction can be started and stopped with any accuracy. Thus an indicated reaction time of 30 sec., may mean that the reaction is complete in less time.

Figure 2 (*b*) shows that a 20 per cent excess of reagent almost doubles the rate of reaction. The reaction is now complete in 7 min. at 25 C. It is practically complete in 30 sec. at 55 C. At 70 and 85 C. it is too rapid to follow.

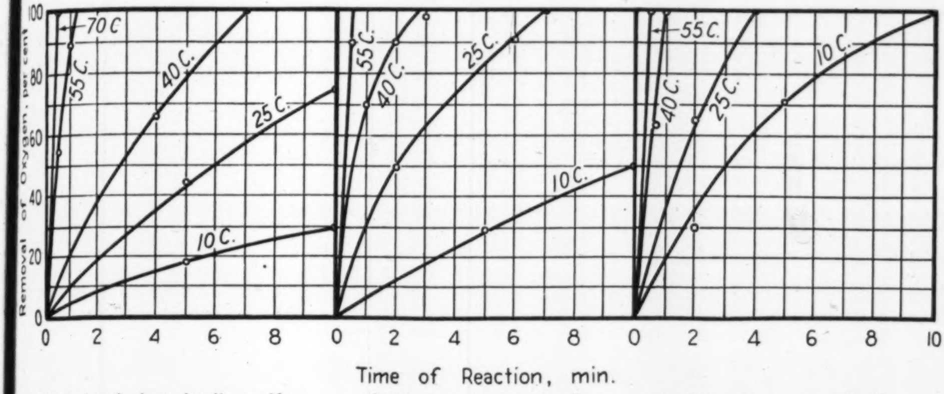
Figure 2 (*c*) shows that addition of a 100 per cent excess of reagent results in another substantial increase in the rate of reaction. At 10 C. the reaction is complete in 10 min., at 40 C. it is complete in 1 min., and at 55 C. it is complete in less than 30 sec.

The accuracy of these determinations is about ± 2 per cent up to 40 C., ± 3 per cent at 55 and 70 C. and ± 4 per cent at 85 C.

The results obtained using St. Louis city water, chosen for its marked tendency to inhibit the reaction, are shown in Fig. 3. Figure 3 (*a*) gives the results obtained with the addition of the theoretical quantity of sodium sulfite. The reaction is extremely slow at 25 C., slow even at 40 C., begins to speed up distinctly at 55 C., is reasonably rapid at 70 C. and is complete in 2 min. at 85 C. Apparently it is only about one-tenth as rapid as in distilled water under the same conditions.

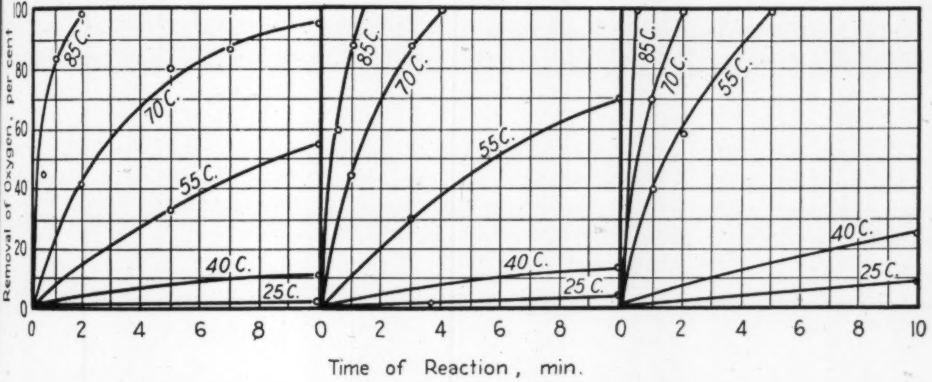
Figure 3 (*b*) shows that with a 20 per cent excess of sodium sulfite the reaction is still slow at 25 and 40 C. The rate increases markedly at 55 C., at 70 C. the reaction is complete in 4 min., and at 85 C. it is complete in a little over 1 min. Although the excess of reagent apparently exerts little effect at the lower temperatures it causes an appreciable increase in the rate of reaction at 70 and 85 C.

Figure 3 (*c*) shows that even with a 100 per cent excess of reagent the reaction rate is slow at 25 and 40 C. At 55 C. reaction is complete in 5 min. At 70 C. it is complete in 2 min and at 85 C. in 30 sec. Thus even in a water which shows strong inhibitory tendencies the reaction still proceeds rapidly at 70 and 85 C. when an excess of sodium sulfite is used.



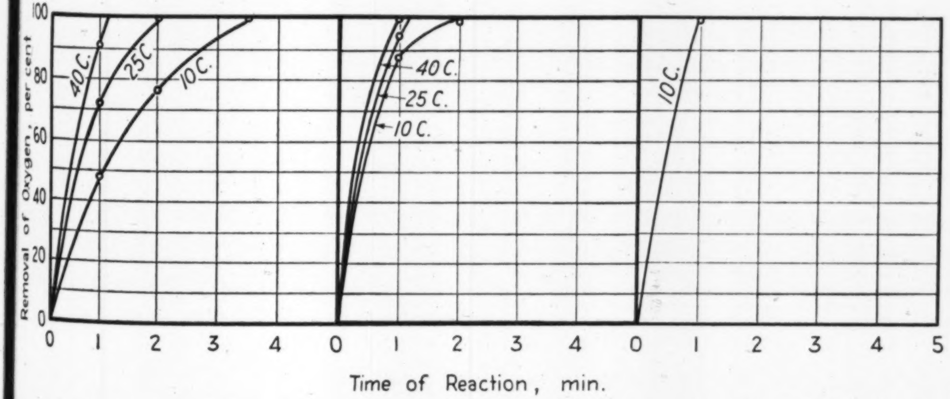
(*a*) One equivalent of sodium sulfite. (*b*) 20 per cent excess of sodium sulfite. (*c*) 100 per cent excess of sodium sulfite.

FIG. 2.—Results Obtained Using Distilled Water. pH value, 7 to 8.



(*a*) One equivalent of sodium sulfite. (*b*) 20 per cent excess of sodium sulfite. (*c*) 100 per cent excess of sodium sulfite.

FIG. 3.—Results Obtained Using St. Louis City Water. pH value, 9.0.



(*a*) One equivalent of sodium sulfite. (*b*) 20 per cent excess of sodium sulfite. (*c*) 100 per cent excess of sodium sulfite.

FIG. 4.—Results Obtained Using Sea Water. pH value, 8.

The data obtained using water taken from the Atlantic Ocean outside of Boston, Mass., are given in Fig. 4. It will be observed that the reaction is so rapid in sea water that the temperature scale has been doubled in plotting the data. Figure 4 (*a*) shows the results obtained with the addition of one equivalent of sodium sulfite per equivalent of oxygen. Even at 10 C. the reaction is complete in 4 min., at 25 C. in 2 min. and at 40 C. in slightly more than 1 min. The reaction is over five times as fast in sea water as in distilled water.

Figure 4 (*b*) shows that with a 20 per cent excess of sodium sulfite the reaction is complete in 2 min. at 10 C. and in 1 min. at 25 C. Figure 4 (*c*) shows that in sea water with a 100 per cent excess of sodium sulfite present the reaction is complete in 1 min. at 10 C. The experiments on sea water were among the first carried out. In that stage of the investigation the technique had not been developed sufficiently to permit the observation of reaction periods of less than 1 min. It is, however, obvious from the results that the reaction must be almost instantaneous under some of the conditions studied.

SUMMARY AND CONCLUSIONS

This investigation of the reaction between sodium sulfite and dissolved oxygen shows that the reaction rate is a function first of the type of water, second of the temperature and third of the excess of the reagent employed. The reaction proceeds roughly five times as rapidly in sea water as in distilled water and ten times as rapidly in distilled water as in St. Louis city water. The reaction proceeds with great rapidity at 85 C. even in water with strong inhibitory tendencies. In general, a rise of 10 deg. Cent. doubles the speed of the reaction. A 20 per cent excess of reagent almost doubles the rate of reaction and a 100 per cent excess almost quadruples it.

It is felt that this investigation, carried out on waters of divergent properties over a wide temperature range and with various proportions of sodium sulfite, lends support to the belief now prevalent among many engineers that sodium sulfite is an effective means for the removal of oxygen from water.

This investigation gives more information about the speed of the reaction than about its completeness because it was necessary to determine the oxygen remaining in the water by an indirect method. In the present work this indirect method has been developed as far as is practicable. If it were possible to develop an extremely sensitive method of determining dissolved oxygen directly which could be applied to water immediately at the expiration of a reaction period, it might be possible to demonstrate more conclusively the completeness of the removal of dissolved oxygen. Perhaps a colorimetric method, such as that described by Pettingill and Barlow,[11] might be applicable.

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[11] C. Pettingill and E. J. Barlow, "Dissolved Oxygen Recorder," *Power*, Vol. 77, p. 63 (1933)

DISCUSSION

Mr. F. N. Speller.[1]—There is no question about the utility of sodium sulfite for removal of oxygen from water at normal temperatures. The paper by Messrs. Hitchens and Towne presents useful data in this connection. We have experimented with sodium sulfite in our laboratory in the treatment of mud-laden fluids used in rotary drilling of oil wells to prevent corrosion-fatigue. We have found that it increased the life of the metal subject to corrosion-fatigue in corrosive muds four or five times. However, we have not been able to determine the exact amount of sulfite or its rate of reaction in these muds when they contain a considerable amount of soluble salts, mainly chlorides. We would like to know whether the authors can make any suggestions on these points which will aid in making these determinations.

In the paper by Schroeder, Berk and Partridge[2] reference is made to the dissociation of sodium sulfite at boiler temperatures. If the sulfite acts as an oxidizing agent in steam boilers, like the chromates, by forming a protective film on the metal as well as affording protection from corrosion by the removal of free oxygen from solution, this opens the way to another principle which may find wide application in the protection of boilers at high temperatures. If a protective film can be maintained on the surface of the metal by reaction between the metal and the water, and can be controlled by proper water treatment, the results may be of great practical importance because one of the problems at present in high-pressure boiler work is to prevent corrosion, especially in the form of pitting. It does not seem to be altogether certain that under these conditions corrosion is entirely prevented by the removal of oxygen. Something more may be required.

However, in laying down protective films of this kind, it is important to remember that most of these films are cathodic to the metal, so that if they are not self-healing and the conditions are unfavorable to maintaining a continuous film, they may accentuate and stimulate pitting. I believe that a study of film formation at these high temperatures will be highly useful. We have found that a comparatively small amount of colloids has a marked effect in reducing corrosion, particularly the form of pitting. This property can probably be utilized to advantage in protecting the internal surface of boilers in contact with the water. Any such treatment, of course, requires careful control to be effective.

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[1] Director, Department of Metallurgy and Research, National Tube Co., Pittsburgh, Pa.

[2] W. C. Schroeder, A. A. Berk and Everett P. Partridge, "Effect of Solution Composition on the Failure of Boiler Steel Under Static Stress at 250 C.," see p. 721.

We have studied film formation in our laboratory to a considerable extent with respect to its effect on the potential of the metal and rate of corrosion and have found that the compound films, such as those formed by phosphates and chromates in water, are much more effective than either one separately.

MESSRS. R. M. HITCHENS[3] AND R. W. TOWNE[3] (*authors' closure by letter*).—The determination of the rate of reaction of sodium sulfite with dissolved oxygen in mud-laden fluids used in rotary drilling of oil wells would be a most difficult task. Even without the physical difficulties imposed by the mud, the rate of reaction experiments are none too easy.

Perhaps a method might be worked out for the approximate determination of the sulfite present. Such a method would depend upon the properties of the mud. We should be glad to cooperate in the development of such a method.

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[3] Assistant Chief Analyst, and Chief Analyst, respectively, Analytical Laboratories, Monsanto Chemical Co. St. Louis, Mo.

DETERMINATION OF DISSOLVED OXYGEN IN BOILER FEED WATER[1]

By Alfred H. White,[2] Claude H. Leland,[3] and Dale W. Button[3]

Synopsis

The present paper constitutes another progress report covering the work being carried out at the University of Michigan under the auspices of Subcommittee VIII on Standardization of Water Analysis Methods of the Joint Research Committee on Boiler Feed Water Studies. The first aim was to establish and evaluate the inherent errors of the Winkler procedure in its application to the determination of concentrations less than 0.10 ml. of dissolved oxygen per liter of water in boiler feed waters. The second aim was the development of a more accurate method for use as a referee procedure in measuring the concentration of oxygen in feed water and in the deaerator effluent. Earlier progress reports (**26, 28, 33**)[4] prepared by the subcommittee have pointed out inherent errors in the Winkler procedure. They discussed the sensitivity of the starch-iodine reaction, the effect of purity of reagents and, in a preliminary manner, the effect of certain ions in the water upon the accuracy of the determination.

The present paper reviews briefly the past work in reference to the effect of certain ions upon the accuracy of the determinations, and the errors which may arise in the titration of the sample. It further presents a study of an electrometric method of titrating free iodine as applied to the Winkler procedure. It is shown that this method of controlling the titration yields results of greater accuracy than those methods where the titration is controlled by a color change. Finally, the results of experimental work are presented which indicate the feasibility of distilling and collecting the dissolved oxygen together with a portion of water distilled from the sample as a method for eliminating non-volatile substances that affect the accuracy of the determination. It is indicated that this procedure may become a referee method for use with waters containing interfering substances.

Review of Earlier Work

The direct Winkler method has been found to be quite accurate and reliable for the determination of oxygen dissolved in pure water except where extremely small quantities of oxygen are involved, as is the case with boiler feed water which has been passed through a deaerator. Impurities in the water cause the direct titration to be relatively inaccurate when determining low concentrations of oxygen. The impurities most commonly mentioned are nitrite, sulfite, and iron ions, and also organic matter. Many ways have been suggested for overcoming their effect when using the Winkler procedure. Besides the errors introduced by these substances, other errors, chiefly within the control of the analyst are those due to the method of sampling, to impurities in the reagents and to the analytic procedure. Only those errors involved in the method of analysis will be discussed in this paper.

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[1] Submitted as a progress report of Subcommittee VIII on Standardization of Water Analysis Methods to the Joint Research Committee on Boiler Feed Water Studies.

[2] Chairman, Subcommittee VIII on Standardization of Water Analysis Methods; Professor of Chemical Engineering, University of Michigan, Ann Arbor, Mich.

[3] University of Michigan, Ann Arbor, Mich.

[4] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 712.

Nitrites cause difficulty through oxidation of hydriodic acid. As the titration progresses, oxygen absorbed from the air regenerates the nitrous acid which in turn liberates more iodine, so that it is impossible to obtain a distinct end point in the titration with sodium thiosulfate. Winkler (10, 12) has shown that interference may be caused by the nitrite ion even when its quantity was as low as one part in ten million. Hale and Melia (8) place the point of interference at two parts in ten million. Von Noll (14), Cronheim (6) and Elvore (9) also considered the interference of this ion. The Rideal-Stewart (4) modification eliminated the nitrites by the addition of potassium permanganate solution, with the reduction of excess permanganate with oxalic acid. This method, although acceptable when determining high concentrations of dissolved oxygen, introduced additional errors large enough to be important when low concentrations of dissolved oxygen were being determined. The principal error was due to the dissolved oxygen introduced with the rather large volume of reagents. The correction for the dissolved oxygen added in the reagents came sometimes to be greater than the amount of oxygen dissolved in the sample of water.

Sulfites are oxidized to sulfates in the Winkler method and cause low values for oxygen. The Rideal-Stewart (4) modification has been applied to water containing sulfites, but the method has proved to be unsatisfactory because of an equilibrium reaction. Von Noll (5), Lunge (7), Winkler (10) and Haase (23), suggested that sulfite be oxidized to sulfate by the addition of iodine. Alsterberg (20) recommended bromine. Winkler (10) and Bruhns (11) considered the conversion of excess manganous hydroxide to carbonate. Winkler stated that the use of calcium hypochlorite (12) and manganic chloride (1) as oxidizing agents was unsuccessful. Wesley (25) considered the separation of manganous hydroxide from the solution in a nitrogen atmosphere to be an improvement. Theriault and McNamee (24) used standard sodium hypochlorite solution to oxidize sulfites. None of these modifications, however, are considered to be completely successful when low concentrations of dissolved oxygen are to be determined.

The presence of ferric iron in water causes high oxygen values by reacting with hydriodic acid to form free iodine. The reaction does not go to completion, so the simple expedient of correcting the oxygen content for the total amount of iron is not applicable. Fellows, White and Wynkoop (28) suggested on the basis of experimental work. that a correction of 0.01 ml.

per liter be subtracted from the oxygen content of the sample when ferric iron was present in an amount from 0.5 to 7.0 p.p.m. Urbain and Miller (21) worked out a curve for the corrections to be applied for various concentrations of iron. Winkler (12) suggested that the sulfuric acid be replaced by phosphoric acid. Bruhns (13) used inert iron oxalates without success. Buswell and Gallagher (16) used hydrochloric acid. They also applied the Rideal-Stewart modification. As a conclusion to their investigation they recommended Letts and Blake's (3) modification of the Levy (2) method.

Organic matter may cause low and erratic values for oxygen. The Rideal-Stewart modification has also been proposed to remove the organic matter, but its use is accompanied by the same inherent errors that have been already discussed.

The use of starch as an indicator may give rise to error. Meisenberg and Fellows (26) showed that the sensitivity of the starch varied considerably with temperature; also, that the sensitivities of various starches were not the same. Yoder and Dresher (31) published results closely checking those of Meisenberg and Fellows. It was established that for greatest accuracy the titration should be carried out at a temperature not above 21 C. using either arrowroot or Baker's c.p. soluble starch as the indicator.

Schwartz and Gurney (29) attempted to offset the effect of impurities in the water and inaccuracies due to the starch end point by treating duplicate samples of water by two procedures. The first sample was treated by the direct Winkler method up to the point where titration with thiosulfate would normally start. Before the titration commenced, a measured amount of biniodate was added to the solution. The second sample was treated with the Winkler reagents in reversed order and then the same quantity of biniodate was added as to the first sample. Both samples were then titrated with thiosulfate using starch as an indicator. The thiosulfate used in the second titration was deducted as a blank from that used in the first titration and the resulting figure was held to give the correct amount of dissolved oxygen.

Prior Work on Electrometric Titration of Iodine:

Electrometric titration has been proposed as a means of determining iodine more accurately than is possible with starch as an indicator. Willard and Fenwick (15) and Van Name and Fenwick (18) have discussed the characteristics of electrometric titrations which use bimetallic electrodes. Foulk and Bowden (19) have applied the bimetallic electrode system to iodometry in a type of titration known as the "dead-stop end point." Hewson and Rees (34) have adapted this "dead-stop end point" titration to the determination of iodine liberated by the Winkler reagents. They conclude from their work that iodine concentrations equivalent to 0.001 ml. per liter of oxygen can be determined with a high degree of probability. Another development in the electrometric titration of iodine liberated by the Winkler reagents has been made by The Leeds & Northrup Co. (35). Their work has not been published but has been communicated to us and has been helpful to us. Permission has been given by Leeds & Northrup to include a summarized description of their method in this paper. Their apparatus consists of a 1 by 1-cm. sheet of platinum, used as an indicator electrode, a saturated calomel half-cell (L & N No. 7724–6), used as a reference electrode, a portable potentiometer (L & N No. 7655), a laboratory stirrer, a fine-tipped burette, and an 800-ml. beaker. The electrodes are mounted in the 800-ml. beaker on a stand in such a manner that the beaker may be easily removed for cleaning after each titration. The normal potential of the iodide-iodine electrode is approximately 0.535 v. while that of the thiosulfate-tetrathionate electrode is 0.130 v. Since the thiosulfate-tetrathionate electrode is irreversible (22) calculations cannot be made on the change in potential in the vicinity of the end point. Therefore, it is necessary to determine the character of the titration curve experimentally.

On condensate waters, Leeds & Northrup found that it was not necessary to conduct a complete tiration curve. A titration to an end point of 0.3150 v. was found by them to give reproducible results. They observed that the end point tended to drift slowly to higher voltage after the addition of thiosulfate when the end point was approached from the iodine side. At the end point there was no tendency to drift. With an excess of thiosulfate, the electromotive force tended to drift to lower voltages. Furthermore, a 30-mv. change was observed at the end point on the addition of 2 drops of 0.001 *N* thiosulfate. These characteristics made the end point easy to detect—a distinct advantage over the starch colorimetric end point. They also called attention to the pronounced effect of temperature, volume of dilution and personal factor on the starch end point as compared to the minor effect of these variables on the electrometric titration. They stated that these advantages of the electrometric end point increased the accuracy of detection of oxygen in terms of iodine.

EXPERIMENTAL STUDIES

Distillation Method:

The measurement sought is the amount of gaseous oxygen dissolved in the water. It is theoretically possible by boiling the water to drive off all of the oxygen together with some steam, condense the steam and collect the distilled water and oxygen in a receiver. The oxygen will be partly dissolved in the distilled water and be partly in the vapor phase. The apparatus designed for this purpose is shown in Fig. 1. Detailed directions for operation of the method are described later in this paper and at the present time only the broad outlines of the process will be given. The sample of water is collected in flask *A* which is brought to the laboratory completely filled with water and connected to the condenser by rubber tubing. The system from the hydrogen tank through the condenser and flask *C* is filled with pure hydrogen. Part of the water of *A* is discarded through the lower stopcock to give a boiling space in the upper part of the flask and the water is then boiled under somewhat diminished pressure until all of the oxygen has been distilled into flask *C* together with a portion of the water from flask *A*. It is evident that all non-volatile substances will be left in flask *A*. Volatile impurities in boiler feed water are rarely met and if sulfur dioxide or other acid materials should be present in the water, they might be fixed by making the water in flask *A* slightly alkaline.

The effectiveness of this procedure was confirmed by many tests and it was shown that the oxygen was completely boiled off when one-tenth of the water had been distilled. This was confirmed by distilling a second portion of the residual water, which showed itself to be free from oxygen. As a further check, varying amounts of air-saturated water were added to the oxygen-free water through a small burette fused into the distilling flask.

Table I.—Precision of Distillation Method.

Air-Saturated-Water Added to Oxygen-Free Water, ml.	Oxygen Added, Calculated from Winkler Tables, ml.	Oxygen Found in Receiving Flask, Titrated Electrometrically, Corrected for Reagents, ml.
1	0.006	0.005
1	0.006	0.006
1	0.006	0.006
2	0.012	0.011
2	0.012	0.011
3	0.018	0.019
3	0.018	0.017

The results in Table I show that the oxygen added was distilled, collected, and analyzed with an error never greater than 0.001 ml. Analysis made on the water remaining in the distilling flask after the boiling procedure confirmed the conclusion that all oxygen had been removed by the distillation.

Comparison of Direct Winkler Procedure with Distillation Method on Boiler Waters:

In order to compare the two methods on a water high in dissolved solids, samples of water were obtained from a boiler of the University of Michigan heating plant. Two separate samples were taken on each occasion. One sample was taken in a 500-ml. McLean bottle (27) and analyzed by the direct Winkler method. The second and larger sample was analyzed by the distillation method. The sample displaced from the distillation flask by hydrogen just prior to commencing the distillation was tested for interfering substances by adding Winkler reagents in a reverse order, the KOH-KI solution coming first, then the acid, and third the $MnCl_2$. This order of adding the reagents is supposed to prevent any reaction with the oxygen in the sample, but to permit any other oxidizing substance present to liberate iodine, which can then be titrated with thiosulfate. By subtracting this amount of thiosulfate from the amount used in the standard Winkler determination, a close approximation of the oxygen in the water is supposed to result if the same conditions of time and temperature of titration prevail in each case. It has been argued that under the same conditions, even though the reactions that cause interference do not go to completion, the rates of reaction should be the same and hence, the extent of the reactions the same.

TABLE II.—COMPARISON OF DISTILLATION METHOD AND DIRECT TITRATION ON BOILER WATER CONTAINING INTERFERING SUBSTANCES.

Sample	Volume of Sample, ml.	Oxygen After Correcting for Reagents, ml. per liter			
		Direct Winkler Method (a)	Amount of Positive Interference Calculated as Oxygen (b)	Apparent Value (a-b)	Distillation Method
No. 1 (*a*)......	500	0.074	0.038	0.036	
No. 1 (*b*)......	870				0.022
No. 2 (*a*)......	500	0.062	0.038	0.024	
No. 2 (*b*)......	865				0.021
No. 3 (*a*)......	543	0.070	0.027	0.043	
No. 3 (*b*)......	1082				0.059
No. 4 (*a*)......	500	0.074	0.035	0.039	
No. 4 (*b*)......	885				0.024
No. 5 (*a*)......	550	0.065	0.000	0.065	
No. 5 (*b*)......	1055				0.067
No. 6 (*a*)......	550	0.035	0.019	0.016	
No. 6 (*b*)......	885				0.037

TABLE III.—EFFECT OF VARYING CONCENTRATIONS OF NEGATIVE IONS ON THE END POINT IN THE WINKLER METHOD.

Concentration of Ions in Iodine Solution, g. per liter				Voltage at End Point	Thiosulphate 0.002 *N* Required to Reach End Point, ml.
Iodide	Chloride	Sulfate	Hydrogen		
0.38	....	....	.....	0.2722	1.64
0.96	....	....	.....	0.2645	1.64
1.91	....	....	.....	0.2300	1.64
0.38	2.07	....	0.058	0.2700	1.92
0.38	5.18	....	0.146	0.2719	2.16
0.38	10.40	....	0.292	0.2705	2.40
0.96	0.24	....	.....	0.2610	1.70
0.96	0.72	....	.....	0.2605	1.66
0.96	0.96	....	.....	0.2640	1.68
0.96	....	0.27	.....	0.2665	1.68
0.96	....	0.68	.....	0.2620	1.64
0.96	....	1.37	.....	0.2645	1.62
1.72	1.15	8.50	0.001	0.2600	1.68
0.12	....	0.85	0.005	0.2935	1.68
0.29	....	2.18	0.012	0.2908	1.71
0.57	....	4.27	0.026	0.2857	1.74
0.57	0.57	4.27	0.026	0.2840	1.73
0.57	0.57	5.15	0.044	0.2860	1.83
1.15	....	8.50	0.053	0.2590	1.87
1.15	1.15	8.50	0.053	0.2568	1.85
0.57	5.18	....	0.083	0.2808	1.98

In a similar way the amount of reducing substances is supposed to be obtained by noting the amount of biniodate required to produce the first trace of iodine. By adding the equivalent amount of biniodate to the thiosulfate used in the standard Winkler determination, it was argued that the resulting oxygen value should be reasonably close to the true value if the same conditions prevail in the titrations. The various columns of Table II show the results of the direct Winkler method, the positive interference found in the water calculated as oxygen, the corrected results of direct procedure, and results of the distillation method. It is noted in samples Nos. 5 (*a*) and 5 (*b*) no interfering substances were found and the two methods check to 0.002 ml. per liter. In the other samples the variations from the distillation method are larger but are not systematic. In three of the samples the direct method gives high results, the maximum being 0.015 ml. per liter and in two of the samples the direct method is low, the maximum variation being 0.021 ml. per liter. These figures indicate that the method of determining interference by adding the reagents in the reverse order does not give reliable results, when compared with the distillation method.

The Electrometric Titration:

In the study of the electrometric titration curve, the thiosulfate - electromotive force relationship was observed in the presence of various concentrations of iodide, chloride, sulfate and hydrogen ions to show the effect of excess of various Winkler reagents on the end point. A series of tests was conducted by titrating 2 ml. of approximately 0.002 *N* iodine solutions in distilled water with 0.002 *N* thiosulfate, temperature and time after adding the reagents being maintained constant. The variations in ion concentrations were obtained by adding small amounts of potassium iodide, chloride, sulfate, hydrochloric acid and Winkler reagents to the solution.

The effect of the concentration of the various ions on both the voltage at the end point and the amount of thiosulfate required to reach the end point is shown in Table III. It is shown that the concentrations of the chloride and sulfate ions have little influence on either the voltage or the amount of thiosulfate required to reach the end point. Increasing concentration of iodide ion causes the end point to occur at lower voltages but does not affect the amount of thiosulfate required to complete the titration. The hydrogen ion concentration, however, has a pronounced influence. As the concentration is increased, more thiosulfate is required to reduce all of the iodine to iodide, the quantity varying from 1.68 ml. thiosulfate with minimum acidity to 2.40 ml. with high acidity. This corresponds to a difference of 0.016 ml. of oxygen per liter when a 500-ml. sample is being titrated. It is probable that the hydrogen ion reacts with the iodide to form hydriodic acid which decomposes in the presence of air to give free iodine. This series of titrations indicates that best results are obtained in the standard Winkler method when the amount of acid used is just sufficient to dissolve the hydroxide precipitate. It is also observed that with low concentrations of iodide ion the end point occurs between 0.280 and 0.290 v., while with more iodide it occurs at 0.230 v. The quantity of thiosulfate is a safer guide than the voltage.

The communication from Leeds & Northrup (35) stated that if the end point was approached from the iodine side, the electromotive force tended to drift to a higher value after the addition of thiosulfate and that if the end point was approached from the thiosulfate side, the tendency was for the voltage to drift to lower values. We have confirmed this observation and also noted that there is a 25 to 35-mv. drop in potential at the end point on the addition of one drop (0.04 ml.) of 0.002 *N* thiosulfate. Hence, it is very easy to detect the end point within ± 0.04 ml. of thiosulfate. This accuracy corresponds to ± 0.001 ml. of oxygen per liter when using a 500-ml. sample.

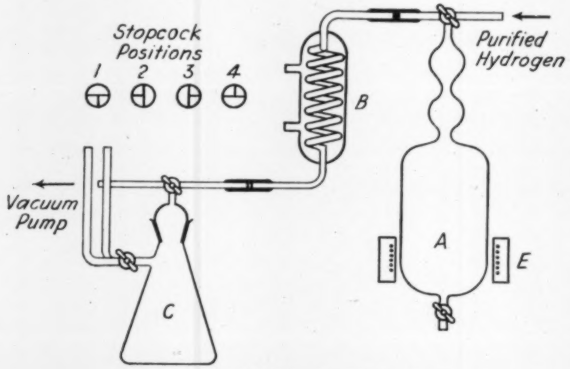

FIG. 1.—Distillation Apparatus.

An even sharper titration curve is produced by adding an excess of thiosulfate and titrating back with an oxidizing agent such as biniodate.

Figure 2 curve 1 represents a typical titration curve produced when titrating iodine with thiosulfate. Curve 2 is the characteristic oxidation curve produced by titrating an excess thiosulfate with potassium biniodate. It is observed that the oxidation curve has a slightly sharper break at the end point than the reduction curve. However, the break in the thiosulfate titration curve is sharp enough to allow easy detection of the end point by a single drop of 0.002 *N* thiosulfate and there appears no need of complicating the procedure by the double titration.

Curve 3 represents the curve produced when titrating the sample obtained from the distillation method. In this case, it is noticed that there is an extremely sharp end point, one drop of 0.002 *N* thiosulfate being sufficient to reverse the potential. The reversal of potential is apparently caused by hydrogen which had been dissolved in the water during the distillation procedure. It has been shown in Table I that this dissolved hydrogen does not have any effect on the amount of thiosulfate required to complete the titration.

Iodide solutions are slowly oxidized by air liberating iodine and the end point of a determination is dependent on the time that a sample is exposed to the air after the Winkler reagents are added and until the titration is completed. Table IV indicates that with increased duration of exposure to air during titration the amount of thiosulfate, and hence the oxygen reported, increases, while in the biniodate titration the amount of biniodate and hence the oxygen reported decreases with time. The difference in results between 10 and 30 min. corresponds to an error of 0.002 ml. of oxygen per liter when a sample of 500 ml. is titrated.

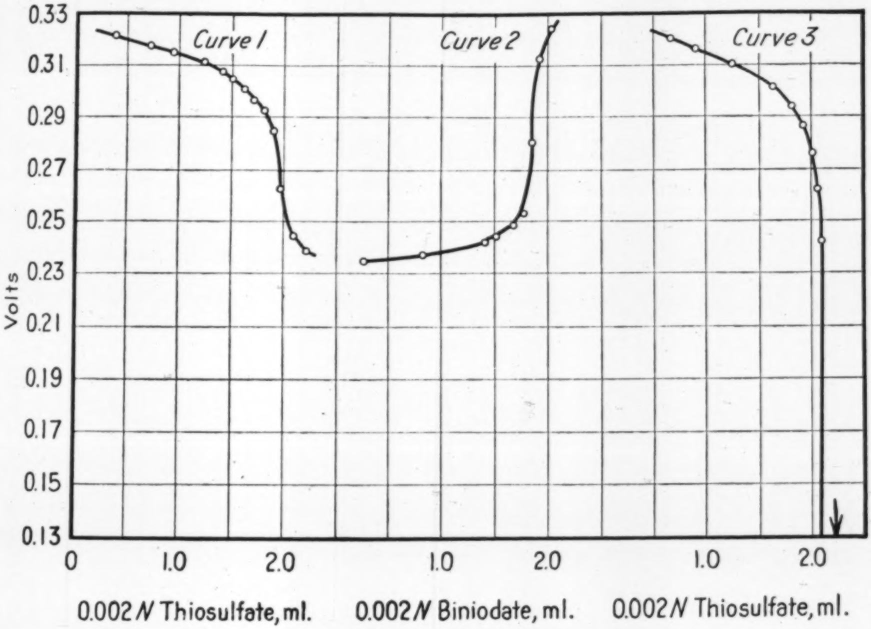

FIG. 2.—Titration Curves.

Correction for Winkler Reagents:

Considerable study by Meisenberg and Fellows (26) in this laboratory indicated the errors caused by the Winkler reagents themselves in an oxygen determination. Table V gives the average values as obtained by direct titration of samples of distilled water freed from oxygen to which various amounts of air-saturated reagents had been added. The titrations using arrowroot starch as indicator are by Meisenberg. The recent values obtained by electrometric titrations are usually 0.001 ml. of oxygen higher than those with starch as an indicator and the small differences between them are due to the lack of sensitivity of the starch as noted by Meisenberg.

TABLE IV.—VARIATION IN ELECTROMETRIC TITRATION DUE TO OXIDATION OF SOLUTION BY CONTACT WITH AIR.

TIME TO REACH END POINT, MIN.	THIOSULFATE REQUIRED TO TITRATE 2 ML. BINIODATE, ML.	BINIODATE REQUIRED TO TITRATE 2 ML. THIOSULFATE, ML.
10	2.08	1.96
20	2.12	1.92
30	2.16	1.86

TABLE V.—CORRECTIONS FOR WINKLER REAGENTS.

Air-saturated reagents added to water free from oxygen.

KOH-KI, ML.	$MnCl_2$, ML.	H_2SO_4, ML.	AVERAGE OXYGEN OBSERVED, ML.	
			STARCH INDICATOR	ELECTROMETRIC TITRATION
1	1	1	0.0015	
1	1	1		0.002
1	2	1	0.003	
2	2	1	0.003	
2	2	2	0.005	
2	2	2		0.006
3	1	3	0.004	
3	2	2	0.005	
3	2	2		0.006
3	3	3	0.008	
3	3	3		0.009
4	1	4	0.005	
4	2	4	0.008	
4	4	4	0.012	

TABLE VI.—OXYGEN DISSOLVED IN WINKLER REAGENTS.

Determined by distillation method.

	SAMPLE, ML.	OXYGEN OBSERVED, ML.	OXYGEN CORRECTED FOR REAGENTS, ML.		OXYGEN CALCULATED, ML. PER LITER
Alkaline potassium iodide	40	0.009	0.003		0.075
	50	0.011	0.005		0.100
				Average	0.090
Sulfuric acid	50	0.007	0.001		0.020
	50	0.009	0.003		0.060
				Average	0.040
Manganous chloride	50	0.090	0.084		1.68
	50	0.088	0.082		1.64
				Average	1.66

A determination of the amount of dissolved oxygen in each of the Winkler reagents was performed using the distillation method. Oxygen-free water was prepared in the distilling flask, the reagent to be tested was added to the flask, and the oxygen distilled and collected in the receiving flask for the determination. The distillation method permitted relatively large samples of each reagent to be tested, with a corresponding gain in accuracy over the previous tests. The oxygen dissolved in each reagent is reported in Table VI. The averaged results from Table VI are given below:

Reagent	Dissolved Oxygen, ml. per. milliliter of reagent
Alkaline potassium iodide	0.00009
Sulfuric acid	0.00004
Manganous chloride	0.00166

The values obtained are in close agreement with the observations of Theriault (30).

In the Winkler procedure adopted by us 3 ml. of KOH-KI, 2 ml. $MnCl_2$ and 2 ml. of H_2SO_4 were employed in each sample in order to maintain a low concentration of hydrogen ion. The oxygen dissolved in these reagents added should be 0.004 ml., from the data given above, taken from Table VI. Actual determinations on distilled water free from oxygen indicated, Table V, that the value of the correction to be applied was 0.006 ml. of oxygen. It is apparent that the correction in actual practice even with the electrometric titration is slightly larger than that due to the oxygen dissolved in the reagents. This discrepancy is at least partly caused by the oxidation of the iodide solution to iodine during the titration.

TABLE VII.—COMPARISON OF DIRECT TITRATIONS ON BOILER FEED WATER APPARENTLY FREE FROM INTERFERING SUBSTANCES.

Sample	Sample, ml.	Temperature, deg. Cent.	Oxygen Corrected for Reagents, ml. per liter	
			Starch Indicator	Electrometric Titration
No. 1	250	25	0.056	
	300	25		0.066
No. 2	250	24	0.094	
	250	24		0.103
No. 3	305	23	0.184	
	240	23		0.185
No. 4	250	25	0.156	
	250	25		0.165
No. 5	240	19	0.148	
	300	19		0.158
No. 6	250	22	0.246	
	250	22		0.252
No. 7	275	16	0.103	
	265	16		0.108
No. 8	275	20	0.080	
	266	20		0.086

Electrometric Titration Compared with Starch as an Indicator on Boiler Feed Water Titrations:

It has been shown that the electrometric titration gives close agreement with the starch-indicator method when tested on distilled water.

It has also been shown that it is not possible to make accurate corrections for the direct titration when there were material amounts of interfering ions present. The tests now presented were made to see what the comparison would be on boiler feed water of good quality which apparently did not contain interfering substances. The results of tests on eight samples as given in Table VII show that the results with starch are always low, usually 0.006 to 0.010 ml. oxygen per liter. The difference is in part due to the lack of sensitivity of the starch but is too large to be charged entirely to this source. It seems probable that the water carries small amounts of interfering substances not detectable by the method of adding the reagents in reverse order.

METHOD OF SAMPLING AND ANALYSIS RECOMMENDED FOR WATERS CONTAINING INTERFERING SUBSTANCES

This method is based on the fact that all the oxygen which is dissolved in a water sample may be distilled with a fraction of the water and collected in a flask where the standard Winkler procedure may be applied. It separates the oxygen from any non-volatile impurities that interfere in the Winkler test and also permits the concentration of oxygen from a large sample. It is necessary to carry out this procedure in an entirely enclosed system and in an inert atmosphere. There are, therefore, three important precautions to be observed.

1. All oxygen must be exhausted from the system before an analysis is made.

2. Sufficient water must be distilled to insure complete removal of the oxygen from the sample.

3. All oxygen boiled out must be collected in the receiving flask where it will react with the Winkler reagents.

Apparatus:

A sketch of the apparatus is shown in Fig. 1. The sample flask *A* was constructed to have a volume of approximately 1200 ml. Two entrainment bulbs in series, each of approximately 50-ml. capacity, are joined to the top of the flask. A stopcock at the bottom and a three-way stopcock at the top permit water to pass through the flask to flush it thoroughly. The water-cooled condenser *B* is of glass and the coil is made of tubing of 3 mm. internal diameter which is small enough to cause slugs of liquid to form and thus prevent oxygen from diffusing back against the flow of vapor. The receiving flask *C* is a 500-ml. Erlenmeyer flask fitted with a ground-glass bonnet and a three-way stopcock to facilitate the flushing of the system with purified hydrogen. Two tubes each holding 4 ml. are fused in the side of the flask for the introduction of reagents. The sample flask, condenser, and receiving flask are joined to one another and to the hydrogen header by short lengths of rubber tubing. The sample flask is heated by an electrical heating element placed around the flask near the bottom. This method of heating has been found to produce steady boiling without bumping, even after the water becomes entirely free of dissolved gases. The chromel resistance heating element contains enough resistance to limit the current from a 110-v. a.c. circuit to 3.2 to 3.5 amp. An external resistance, not shown, serves to control the rate of boiling.

Sampling Boiler Feed Water:

The sample flask *A* is disconnected from the remainder of the apparatus, placed in a carrying case and transported to the source of supply of the boiler feed water. The method employed in obtaining the sample is extremely important for if the sample is not fully representative of the water to be analyzed there will be no advantage in the use of a precise method of analysis. The following method is based on that recommended by the Cochrane Corp. (32).

A $\frac{1}{8}$-in. opening is provided at some convenient point in the line carrying the water to be tested. The connection consists of a short nipple and a stop valve to which a coil is attached with a needle valve on the discharge side of the coil. The coil is to be placed in a container into which cold water may be run continuously while samples are being taken. It is important to observe the next steps:

1. Have the stop valve ahead of the cooling coil fully open while samples are being taken.
2. Flush out the system by opening the needle valve fully and allowing the water to run to waste for several minutes.
3. Adjust the needle valve so that the water leaves the condenser at no more than room temperature and allow the water to run to waste for 10 min.
4. Attach the cooling coil to the bottom cock of the sampling flask by means of a short piece of rubber tubing and allow the flask to fill and overflow until its contents have been displaced at least five times.
5. Close all stopcocks and remove the flask to the laboratory for analysis.

DISTILLATION METHOD OF ANALYSIS

In the laboratory the sample flask, completely filled with water, is connected to the hydrogen line and to the condenser as shown in Fig. 1. It is not safe to assume that hydrogen from cylinders is free from oxygen and so a furnace containing a tube filled with platinized asbestos should be placed in the hydrogen line. The analysis should not be started until this tube has been heated to 425 C. for 10 min. At this time it is convenient to introduce 3 ml. of alkaline potassium iodide reagent into one of the side tubes of flask *C*, and 2 ml. of manganous chloride into the other. The cock on *C* is then changed to position 1 and the system is evacuated. The cock is next changed to position 3 and the flask filled with hydrogen. This process should be repeated five times to remove all oxygen in the condenser and receiver. It is desirable to install a mercury manometer in the hydrogen line so that the pressure on the system may be known at all times. After the last evacuation, the pressure of hydrogen is allowed to increase until the mercury gage shows about 2 in. of positive pressure. The three-way stopcock on *A* is then turned to position 3 so that connection is established between the sample flask and the hydrogen supply. The bottom stopcock of *A* is then opened and, due to the slight gas pressure, water will be forced out of *A* into a graduated cylinder. The flow should be stopped and the supply of hydrogen turned off when the water level is about 1 in. below the lower of the two bulbs in the upper part of *A*. The volume of the final sample is obtained by deducting the volume of water in the graduate from that of *A* when full. The cock on *A* is then returned to position 4 and that on *C* to position 1 and a final vacuum of about 18 to 20 in. of mercury is produced in flask *C*. Stopcock on flask *C* is then turned to position 3, and on flask *A* to position 2 so that the pressure is equalized. There will now be a partial vacuum of 8 to 10 in. of mercury in the system so that there will be no danger of blowing up the flask during the distillation .

Heat is then applied to the sample by means of the heating coil in order to boil out the dissolved oxygen and distill over a portion of the sample. During the distillation, care must be exercised to see that the stopcock on the top of the sample flask does not get so hot as to cause the lubricant to flow. This would permit leakage of air into the receiving flask which is under a slight vacuum and thus destroy the value of the determination. An effective, though crude, method of keeping the stopcock cool is to wrap a wet rag around it.

The reagents to be used are substantially those recommended by others:

1. Alkaline potassium iodide. 700 g. KOH and 150 g. KI dissolved and diluted to 1 liter.

2. Manganous chloride. 412 g. $MnCl_2 \cdot 4H_2O$ dissolved and diluted to 1 liter.

3. Sulfuric acid. Concentrated acid, specific gravity 1.84, diluted with an equal volume of water.

After the first 15 to 20 ml. of distillate has been boiled off, the 3 ml. of alkaline potassium iodide and 2 ml. of manganous chloride already in the side tubes are added to the contents of the receiving flask. The receiving flask is shaken intermittently during the distillation for a total time of 5 to 8 min. so that the precipitate of manganous hydroxide will react with any oxygen that may be in the vapor space. When 100 to 150 ml. of the sample has been distilled into the receiving flask, 2 ml. of sulfuric acid are added to the contents of the flask through the manganous chloride side tube. The flask is then shaken until all the precipitate has dissolved. If, however, the vacuum on the system at the beginning of the distillation was not enough, the pressure on the inside of the flask at this time may be higher than atmospheric. If acid cannot be introduced by blowing it into the flask under a very slight pressure the determination will be lost.

Electrometric Titration:

After the acid has been added and the precipitate dissolved, the flask can be opened to the air, but this should not be done until the sample is to be titrated. The flask is then emptied into a 400-ml. beaker and rinsed with ordinary distilled water. Titration should be commenced at once by the Leeds & Northrup electrometric method for there will be slow oxidation of the iodide from the distilled water added and from oxygen absorbed from the air. Standard sodium thiosulfate (0.002 N) is added fairly rapidly until the electromotive force is approximately 0.31 v., with stirring prevailing throughout the entire titration. The titration is then conducted drop by drop until the end point is reached. The end point of the reaction occurs between 0.21 and 0.27 v. The character of the curve is shown in curve 3 of Fig. 2. With the addition of the last drop of 0.002 N thiosulfate the voltage drops abruptly from +0.24 v. to a negative value. The time taken to titrate the sample from the moment the receiving flask is opened to the air until the end point of the titration is reached should not exceed 15 min.

CONCLUSIONS

The direct Winkler method for determining dissolved oxygen in water may have an accuracy of 0.001 ml. of oxygen in the volume titrated, if there are no interfering substances in the water and the end point is determined electrometrically. Corrections for interfering substances such as iron, nitrites, and organic matter cannot be made with certainty. Interfering substances, if non-volatile, may be removed by boiling off the dissolved oxygen and collecting it again in distilled water where it is determined by the Winkler method. The oxygen from a 1-liter sample may thus be concentrated to one-tenth of that volume and titrated in a solution freed from all but volatile impurites. Corrections should be made for oxygen introduced with the reagents and for oxidation during exposure to the air during the titration. The corrections are small and capable of accurate determination and the method is theoretically sound and free from systematic errors.

The electrometric method of titration eliminates the error in the starch end point due to inability to decide when the blue color has disappeared, and due to the lack of sensitivity of the starch indicator. The end point in the electrometric titration can be easily detected to within plus or minus 1 drop of 0.002 *N* thiosulfate. One drop from a fine burette corresponds to 0.0005 ml. of oxygen. To ensure the highest accuracy each operator should standardize his own procedure and establish his own corrections. It is believed that if a 1-liter sample is taken for the distillation procedure the over-all error need not be over 0.002 ml. of oxygen per liter of water.

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DISCUSSION

MR. R. C. ADAMS, JR.[1] (*presented in written form*[2]).—The authors of this paper are to be congratulated upon their work which appears to have reduced to a new minimum the probable error in analysis by the basic Winkler method. Certainly laboratory instruments of greater precision must be available before any appreciable improvement in accuracy can be attained.

The applicability of their procedure, however, must be definitely limited because of the large amount of special equipment which the method requires. It is obvious that an ordinary power plant could not provide a furnace for purifying hydrogen, an electrometric titration apparatus, and the other material demanded by the described procedure. Such an outlay might easily double the plant's laboratory investment, as well as demand personnel superior in training to that available at present.

During the past two years the U. S. Navy has become interested in dissolved oxygen in boiler water feed. Since the change in the Navy's method of boiler water treatment, which was made in 1933, all new boilers have been maintained in a scale-free condition and the old boilers gradually are losing the scale deposits which formerly protected the metal against attack by dissolved oxygen. This change in conditions and the resulting possibility of boiler corrosion have caused some concern in the Bureau of Engineering. The Engineering Experiment Station has been requested to survey the existing dissolved-oxygen conditions in the feed water of Naval vessels.

The first effort to obtain the desired data was made on a ship which cruised to Europe during the summer of 1935. All necessary equipment and reagents were supplied by the Experiment Station. The enlisted man who was to perform the analyses, whose previous experience had been limited to routine analyses of boiler water and fuel oil, was instructed thoroughly in a procedure similar to that described by H. S. Lewis in the September, 1934, issue of *Power*. Analytical results covering a period of almost three months were submitted after the cruise. These results were considered to be worthless, because of their inexplicable variations.

A second trial was made, this time by the writer, during the post-repair trials of a Naval vessel in November, 1935. The sampling procedure and equipment were that of Schwartz and Gurney, as described in *Bulletin XXI* of Louisiana State University. The bi-iodate titration was not used, but the free iodine in each sample was titrated in a volume of 500 ml. with 0.01 *N* thiosulfate. The difference between the titrations of the 250 and 500-ml. samples was considered equivalent to the oxygen in 250 ml., since the errors in the two titrations should cancel.

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[2] The opinions of this discussion are those of the writer, and are not to be considered as official opinions either of the Navy Department or of the U. S. Naval Engineering Experiment Station.

It was discovered that a method which has given satisfactory results in a laboratory may be worthless when used on board a destroyer plowing through a seaway at full power. Ordinary apparatus slithered about on the work bench which was the improvised laboratory desk, samples slopped out of beakers during titration, and the swaying burette was difficult to read. The greatest source of error was that reagents could not be added through the third hole in the stopper of a sampling flask without contamination by oxygen from the air.

In the interval before the next analyses were undertaken, several revisions were made in the apparatus for sampling and analysis. The most important change was that in the method for adding the reagents to the sample without exposure to the atmosphere. This was accomplished by the use of specially designed, stoppered-tip pipettes,[3] one of which is shown in the accompanying Fig. 1. Three of these pipettes were fitted through the stopper of each of the wide-mouth Erlenmeyer flasks. In this way the sample could be collected and the reagents necessary to free the iodine and then to acidify and stabilize the solution could be added before the stopper was removed and the surface was exposed to the air. Inaccuracies in the measurement of samples and reagents were eliminated by substituting transfer and measuring pipettes for graduated cylinders and burette.

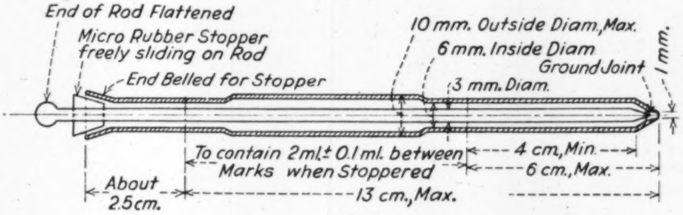

FIG. 1.—Stoppered-Tip Pipette.

The revised apparatus was tested in a more extensive survey of dissolved-oxygen conditions during the builder's trials of another Naval vessel. The results of this survey were sufficiently consistent to indicate that the method requires refinement but no major revision. Complete flushing of air from the sampling train requires twelve to fifteen complete changes of the water so that sampling requires twenty to thirty minutes.

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[3] This design now is subject to investigation by the Judge Advocate General of the Navy in anticipation of an application for U. S. Patent.

This means that an extensive survey within a limited time will necessitate considerable duplication of sampling apparatus and connections.

The most serious deficiency in the procedure is one personal to the analyst. Fellow landsmen may appreciate that the necessity for filling a series of pipettes, particularly 250-ml. pipettes, while using the unsteady footing of a ship's deck, is no aid to continuity of service. This difficulty will be removed by use of an aspirator or a suction bulb on each pipette.

The complete sampling train is illustrated in Fig. 2. It consists of one 250- and two 500-ml. flasks connected in series. Stoppered-tip pipettes are fitted through the stoppers of the first two flasks so that the Winkler reagents may be added before the sample is exposed to the air. The water in the third flask is used for diluting the 250-ml. sample to 500-ml. before titration, and for rinsing the sampling and titration flasks in preparation for a subsequent sample.

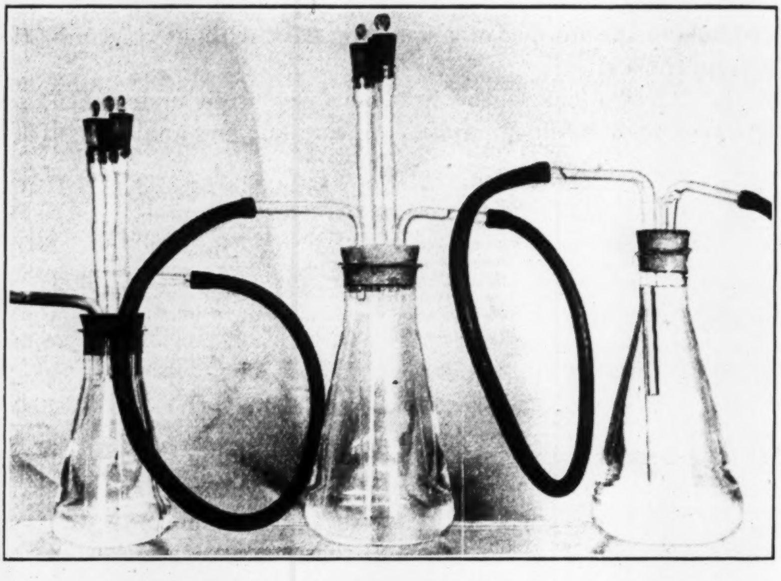

FIG. 2.—Navy Sampling Train for Dissolved Oxygen.

When the sample has been collected, the flasks are removed in the reverse order of their connection. The contents of each flask are protected against absorption of air by looping the discharge tubing around to the inlet glass tube, as shown in Fig. 3. A bubble of air usually is entrapped in the horizontal portion of the inlet tube, but oxygen from this bubble cannot penetrate into the body of the sample. As the reagents are added from the pipettes they displace a portion of the sample, which is allowed to discharge through the inlet tube. After the acid has been added, the alkaline floc has been observed to persist for as long as thirty minutes in the tubing within the flask. Diffusion of oxygen through the tube therefore must be absent.

The most completely deaerated water analyzed by the outlined method contained about 0.07 ml. of oxygen per liter. Results indicate that on such water the accuracy of determinations is about ±0.005 ml. of oxygen. It is believed that such a degree of accuracy is sufficient for virtually all control and investigative needs. An investment of less than fifty dollars in apparatus should provide the means for satisfactory dissolved-oxygen determinations in any plant which has a control laboratory. The method does not require the services of a highly skilled laboratorian.

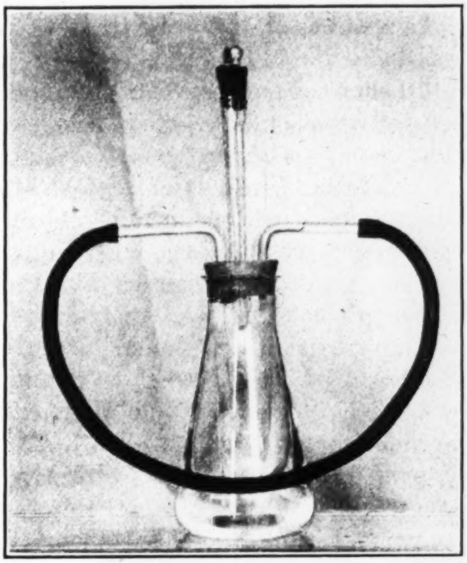

FIG. 3.—Sampling Flask Removed from Train.

MR. T. H. DAUGHERTY[4] (*presented in written form*). The procedure described in the paper appears quite promising for a referee method, but there are a few points that should be investigated further before the work is completed.

In discussing the Schwartz and Gurney method *A*, the objection to it is pointed out that addition of reagents in the reverse order in duplicate samples does not give reliable results, due possibly to incomplete or equilibrium reactions. The Schwartz and Gurney method *B* does not utilize this addition of reagents in reverse order, and hence is not open to this objection. However, it is not discussed here in comparison with the distillation method.

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[4] Research Chemist, Hall Laboratories, Inc., Pittsburgh, Pa.

It would seem desirable that a combination of this distillation and electrometric titration procedure with the Schwartz and Gurney method *B* double titration procedure be investigated. Such a combined procedure should eliminate interference from the principal non-volatile substances, provide for cancelling out the interference due to possible volatile substances, such as Cl_2 and H_2S, increase the accuracy of the titration, and at the same time eliminate the necessity of subtracting a factor for the dissolved oxygen or contaminants in the reagents added.

Also, some studies should be made of the possibility of determining dissolved oxygen in the presence of sulfites by this procedure or by the suggested combined procedure.

Meisenberg's and Fellows' Progress Report of July, 1933, is quoted as recommending either arrowroot or Baker's c.p. soluble starch as the indicator, but instead the root starches, arrowroot and potato, are the ones recommended in the reference. In a later report (May 3, 1935), these authors specify arrowroot starch, with no mention of soluble starch.

MR. R. C. BARDWELL.[5]—As one of the inactive members of the committee who did not have a great deal of contact with the actual progress of the study, it is believed proper to make a few remarks here. At the first reading of the report, my reaction was similar to that which Mr. Adams evidently had when he read it. In other words, the method seemed to be very intricate and very highly technical which would cause possible serious serious problems in properly carrying it out. However, I had the opportunity and pleasure of going over this apparatus with Mr. White in his laboratory, and was very agreeably surprised to find that it was actually relatively simple, once the equipment is set up. The method is comparatively easy to carry out and, with reasonable care, the accuracy seems to be far ahead of former methods. It appears to be very desirable as a final or referee method.

MR. J. R. MCDERMET.[6]—I had the opportunity to read an advance copy of this paper, and as a result, I secured the electrometric titration apparatus and have tried it out in two instances in the field. In making these trials, my viewpoint is necessarily very different from that of the authors. I have been obliged to transport test apparatus and operators in one case for several hundred miles and will continuously face the necessity of doing this in the future. The cost of doing this is considerable. The characteristic, I believe, of any industry in which oxygen titrations are significant is that the apparatus must be transmitted to the equipment under test.

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[5] Superintendent of Water Supply, The Chesapeake & Ohio Railway Co., Richmond, Va

[6] Chief Engineer, Elliott Co., Jeannette, Pa.

It is impossible to transport samples to a remote laboratory under carefully controlled conditions. Frequently also, in approaching a field test, one does not know precisely under what conditions the testing must be done.

In trying out the electrometric end of this titration and entirely disregarding the distillation feature of it, I have utilized it in connection with both the straight Winkler and the Schwartz and Gurney modifications and I have obtained the following observations: In work on water samples, none of which was pure, the method gave surprisingly accurate and consistent results. It apparently will give slightly higher oxygen values than the starch indicator. It was consistent and useful both with the Schwartz and Gurney and the straight Winkler method on samples which were not contaminated enough to upset the regular Winkler titration. It eliminated the question of cooling the samples, which sometimes consumes a lot of time. It was independent of lighting conditions under which the work was performed and this in itself is a very important advantage.

I have, from a personal viewpoint, always been unwilling to take a temperature sensitivity curve for starch indicator because that sensitivity curve involves all the errors one may acquire experimentally and almost always involves a correction for oxygen dissolved in reagents. It is frequently quite a nuisance to locate a supply of ice. The advantage, therefore, of independence of temperature of the sample is frequently very important, particularly when in addition, bad lighting is also involved in the working conditions.

We frequently get dirty samples of water where color is hard to observe, and this electrometric method is almost independent of any coloration of the sample. It, of course, in the straight Winkler method, gives no indication of chemical interference.

As a disadvantage, it requires two operators and one of them must be skilled. I have chosen the alternative of being very careful of the electrode in the matter of cleanliness in its preparation before using. I doubt whether this can be avoided, but if such is the case, the requirement of the skilled operator will probably always persist.

On the distillation method, I have had no experience but I fear again that as outlined, it will be very expensive to transport the apparatus and to use it intelligently, particularly under conditions of light, vibration, etc., such as Mr. Adams has described. From the standpoint of accuracy, the distillation method is certainly of advantage, but I fear that it will have to be simplified further before it will be useful in field work.

MR. BARDWELL.—There seems to be some misunderstanding in regard to the time element required in making dissolved oxygen tests by this method. As I understood it, the time between taking the sample and actually running the test is of small importance. The samples are taken with the usual precautions in the special flask which is filled completely with the water and then sealed. The flask can then be carried without affecting the oxygen content, great distances if desired, to the location of the testing apparatus where the final examination is made. It might be well if this point were clarified.

MR. ALFRED H. WHITE[7] (*author's closure*).—The problem which was set was to devise an accurate method and it is feared that some sacrifice in simplicity is necessary if accuracy is to be attained. It must be recognized that the distillation process does involve somewhat elaborate equipment. However, as Mr. Bardwell has stated, the samples of water can be transferred from the collecting point to the apparatus which may remain set up permanently in the laboratory. We have used collecting cylinders of a liter capacity, with stopcocks at top and bottom. After these have been filled completely with the sample of cooled water they may be set aside and analyzed the next day if desired. We have found that one man can readily make the analyses without assistance. It is pleasant to know that Mr. McDermet has found the electrometric titration satisfactory even under somewhat adverse conditions.

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[7] Chairman, Subcommittee VIII on Standardization of Water Analysis Methods; Professsor of Chemical Engineering, University of Michigan, Ann Arbor, Mich.

EFFECT OF SOLUTION COMPOSITION ON THE FAILURE OF BOILER STEEL UNDER STATIC STRESS AT 250 C.[1]

By W. C. Schroeder, A. A. Berk, and Everett P. Partridge

Synopsis

Observations in practice coupled with the results of laboratory investigations have led to the general opinion that embrittlement failures in boilers are due to the simultaneous action of two factors, an essentially chemical factor depending upon the composition of the boiler water, and an essentially mechanical factor depending upon the stress conditions in the steel. The investigation considered in this paper has been primarily directed to secure information concerning the chemical factors involved in the production and prevention of embrittlement. The tentative conclusions will be subject to modification with further work in the present investigation and the many other investigations now being conducted on the embrittlement of boiler steel. It is believed that a new viewpoint has been offered concerning some of the factors involved in the production and prevention of embrittlement, and of some of the high-temperature reactions which may take place in a boiler water.

The experimental work indicates that chemically pure sodium hydroxide has little effect on the tensile load which a steel specimen will carry, but if the specimen has a line of stress concentration, such as that caused by a groove, chemically pure sodium hydroxide definitely decreases the load-carrying ability. Similarities are shown between hydrogen embrittlement and embrittlement by chemically pure sodium hydroxide solutions. The marked effect of sodium silicate and the influence of sodium sulfate and some other compounds on the embrittlement reactions are discussed. It is also shown that sodium sulfite is reduced by iron and hydrogen at 250 C.

A description of the equipment used in the testing is given first, and this is followed by a discussion of the experimental results approximately in the order indicated in the preceding paragraph. The final summary discusses the conclusions which may be drawn at the present time.

Experimental Procedure and Equipment

The experimental procedure followed in comparing the action of various solutions on boiler flange steel consisted in applying a definite static stress to the steel and measuring the time required to produce failure, or the resistance which the steel offered to failure. While some tests have been run for periods up to 43 days it has been found in general that 10 days is usually sufficient to distinguish accurately between the effect of a solution that will produce a characteristic embrittlement failure and one that will not produce such a failure. The authors recognize the fact that it may be highly desirable to run tests for longer periods but do not believe that it will be advantageous to do so until a more complete understanding is secured of all the factors involved in this method of measuring embrittlement. A second method consisting in the determination of the load a test specimen will carry has been used to compare the action of chemically pure sodium hydroxide solutions with the action of sodium silicate - sodium hydroxide solutions. Both methods of testing have been supplemented by microscopic examination of numerous test specimens.

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[1] Published by permission of the Director, U. S. Bureau of Mines.

This paper has been prepared in connection with an investigation conducted under a cooperative agreement between the Joint Research Committee on Boiler Feed Water Studies and the U. S. Bureau of Mines. The general investigation is supervised by a subcommittee of which J. H. Walker is chairman, and is carried out at the Nonmetallic Minerals Experiment Station of the Bureau of Mines, New Brunswick, N. J.

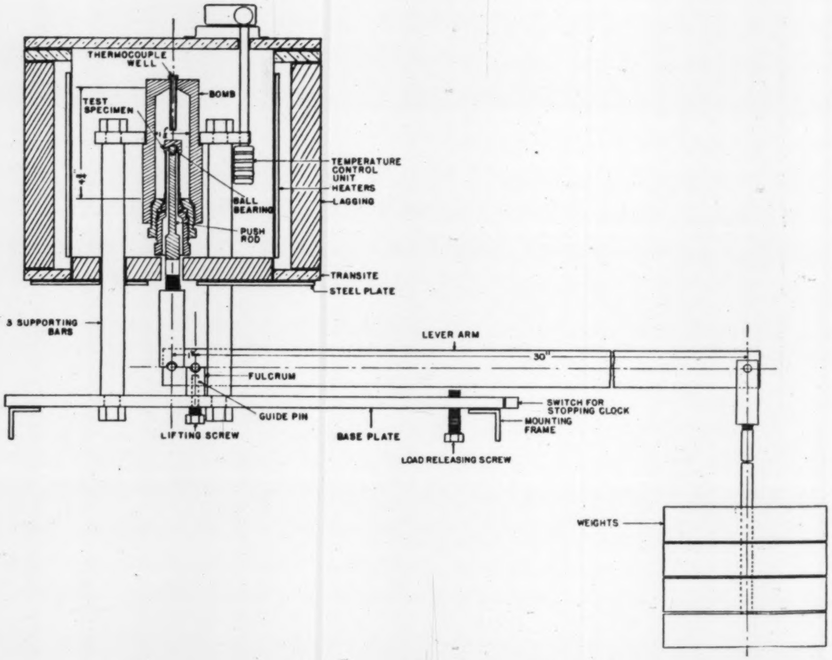
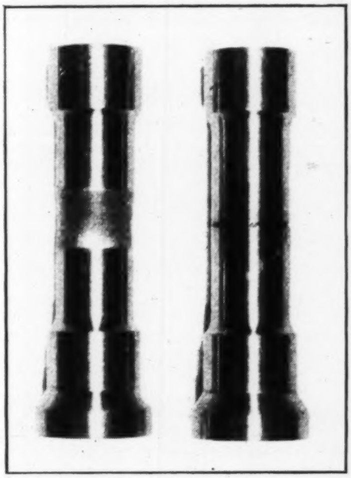

THERMOCOUPLE WELL
BOMB
TEST SPECIMEN
TEMPERATURE CONTROL UNIT
HEATERS
LAGGING
BALL BEARING
PUSH ROD
TRANSITE
STEEL PLATE
3 SUPPORTING BARS
LEVER ARM
30"
1"
FULCRUM
SWITCH FOR STOPPING CLOCK
GUIDE PIN
MOUNTING FRAME
LIFTING SCREW
BASE PLATE
LOAD RELEASING SCREW
WEIGHTS

FIG. 1.—Test Bomb and Loading Mechanism.

The static stress applied to the specimen was either a tensile pull, in which care was taken to secure a very uniform distribution of the load, or a tensile pull combined with bending along a line of stress concentration.

The bomb, the lever system by which the load was applied, and the jacket that acted as an air thermostat are shown in Fig. 1. The temperature of the bomb was controlled by a bimetallic element in the air space between the electrically heated jacket and the bomb. An electric clock controlled by a switch under the lever arm indicated the time of failure for the specimen being tested. Lever systems have been used with 30 to 1, 15 to 1 and 10 to 1 ratios. The lever arm ratio caused no measurable difference in the results.

The test specimen consisted of a tube closed at one end, and the load was applied by means of the rod passing through the center, as shown in Fig. 1. The ball bearing at the upper end of the push rod was inserted to insure the application of the load to the exact center of the head of the specimen. The use of the tubular specimen eliminated the necessity for a packing gland, thus obviating both leakage and the possibility that the gland might exert sufficient friction to cause a serious error in the load actually applied to the specimen. Experience with other types of equipment in which a packing gland was used has shown that the friction due to the gland may vary widely, depending largely on how tight the investigator deems it necessary to pull down the gland to prevent leakage. If a packing gland is used for tests of this type it would be extremely desirable to measure the friction in the packing gland after the completion of each test in order to obtain at least a comparative idea of its magnitude. Calculation will also show that the tubular specimen has a higher ratio of surface exposed to solution to cross-sectional area of metal than a comparable solid specimen and this was considered to be an additional advantage.



Concentric ground. Eccentric grooved.

FIG. 2.—Tubular Specimens Used in Embrittlement Testing.

The two types of test specimen used are shown in Fig. 2. The one indicated as concentric ground was carefully prepared to insure a uniformly distributed tensile load, with a minimum of bending. The reduced section was ground with a fine emery wheel running parallel to the length to eliminate surface scratches perpendicular to the direction of application of the load. These specimens had an inside diameter of 0.500 in., and an outside diameter of 0.620 in. The smallest diameter of the ground section was 0.600 in. and this section was about $\frac{1}{2}$ in. long.

The eccentric-grooved specimen had an inside diameter of 0.500 in., and an outside diameter of 0.630 in. The outer and inner cylindrical surfaces were concentric, but the groove was cut on an axis displaced 0.010 in. from the axis of these surfaces. The maximum depth of the groove was 0.025 in. and the minimum 0.005 in., making the diameter of the base of the groove 0.600 in. This diameter was measured directly by the use of micrometers and two hardened V-shaped blocks with a radius smaller than that of the groove. The radius at the base of the groove was 0.0056 in., and the radius on the tool which cut this groove was compared with a template inserted in the eyepiece of a microscope for every 25 or 30 specimens. For this number of specimens the wear was imperceptible. All average stress values for the eccentric-grooved specimen are calculated for the area of metal at the base of the groove. The deepest section of the groove was always placed in a definite position with respect to the lever arm to maintain as uniform loading conditions as possible.

TABLE I.—AVERAGE PHYSICAL PROPERTIES OF THE BOILER-FLANGE STEEL AT ROOM TEMPERATURE AND 250 C.[a]

Ladle analysis supplied by manufacturer: Carbon 0.17 per cent, manganese 0.47 per cent, phosphorus 0.021 per cent, sulfur 0.039 per cent.

TEMPERATURE	YIELD POINT, LB. PER SQ. IN.	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	REDUCTION OF AREA, PER CENT
Room	28 000	58 000	40	63
250 C	21 000	69 000	33	53

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[a] Tests carried out on Standard A.S.T.M. specimens. Reduced section $\frac{1}{2}$ by $2\frac{1}{4}$ in.

Both specimens had an over-all length of $3\frac{1}{16}$ in. and a reduced section between the heavier ends, which was $1\frac{3}{4}$ in. long. The reamed internal surface was free from scratches in all cases and presented a mirror-like finish to the eye.

The two types of test specimen were used to study somewhat different characteristics of the embrittlement reactions. The concentric-ground specimen was used to investigate the action of the solutions when it was desired to have accurate knowledge concerning the average stress applied to the section of metal exposed to the solution. The grooved specimen gives stress conditions which are representative of those in which embrittlement is found to occur in practice and was used to compare the effect of solutions. Since in this paper the discussion pertains mainly to the comparative action of various solutions it will be found that the grooved specimen was used in most of the tests.

All of the test specimens were machined from a 1-in. plate of boiler-flange steel prepared by the Carnegie Steel Co. The average physical properties of the steel at room temperature and 250 C. are shown in Table I. This table also shows the ladle analysis as furnished by the manufacturer. The layout of the plate from which the specimens were cut is shown in Fig. 3. In the subsequent tables each test specimen has been numbered by a decimal system, the number ahead of the decimal corresponding to the plate from which the specimen was cut and the number after the decimal indicating the position in this plate.

All specimens were cut with their axis parallel to the direction of rolling and all tests were run at 250 C. corresponding to a gage pressure for water of 38 atmospheres (482 F., 561 lb. per sq. in.). The values shown in Fig. 3 for the tensile strength and yield point will serve to give an accurate picture of the variation at room temperature which may be expected in steel taken from different portions of the plate.

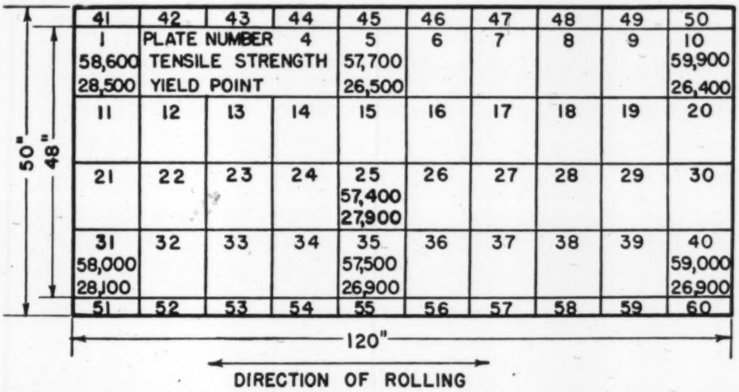
41
42
43
44
45
46
47
48
49
50
1
PLATE NUMBER
4
5
6
7
8
9
10
58,600
TENSILE STRENGTH
57,700
59,900
28,500
YIELD POINT
26,500
26,400
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
57,400
27,900
26
27
28
29
30
31
58,000
28,100
32
33
34
35
57,500
26,900
36
37
38
39
40
59,000
26,900
51
52
53
54
55
56
57
58
59
60
50"
48"
120"
DIRECTION OF ROLLING

Fig. 3.—Layout of Plate and Properties at Room Temperature of the Flange Steel Used in the Embrittlement Tests.

All values average of two specimens. Reduction of area 60 to 64 per cent. Elongation 39 to 42 per cent. Thickness of plate, 1 in. Tensile strength and yield point in pounds per square inch.

Action of Chemically Pure Sodium Hydroxide

From the data available in the literature it would be expected that chemically pure sodium hydroxide would reduce the ability of a steel specimen to carry a uniformly distributed tensile load. The results in this section of the paper indicate that this is not the case but they do show that chemically pure sodium hydroxide will reduce the load-carrying ability of a specimen suffering a stress concentration such as caused by a groove. Data are also presented for the influence of sodium sulfate and potassium chromate on the action of chemically pure sodium hydroxide on the grooved specimens.

Table II.—Short-Time Tests in the Embrittlement Bombs to Determine the Breaking Load for Concentric-Ground Specimens.

Temperature, 250 C.

Specimen	NaOH Concentration, g. per 100 g. H_2O	Breaking Load, lb. per sq. in.	Elongation, per cent[a]
No. 33.17	water	82 700	34
No. 33.28	water	82 100	38
No. 33.19	10	83 100	34
No. 33.20	10	82 000	34
No. 33.16	25	83 300	31
No. 33.17A	25	82 100	28
No. 35.2	50	78 400	22
No. 35.3	50	84 000	25
Average		82 200	

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[a] Calculated arbitrarily on a 0.5-in. length corresponding approximately to reduced section.

Table III.—Resistance to Failure of Concentric-Ground Specimens in Water and Sodium Hydroxide.

Average applied stress, 75,000 lb. per sq. in. Temperature, 250 C.

Specimen	NaOH Concentration, g. per 100 g. H_2O	Duration of Test, days
No. 35.6	water	43
No. 34.16	25	21
No. 35.5	25	43

Table IV.—Short-Time Tests in Embrittlement Bombs to Determine Breaking Load for Eccentric-Grooved Specimens and Load They Will Carry in Water Alone.

All specimens in water. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.[a]	No Failure, days
No. 23.27	73 500	Breaking test
No. 23.28	70 200	
No. 35.17	70 000	
No. 24.16	65 000	15
No. 24.17	65 000	15

---

[a] In this table and in all subsequent tables the average applied stress for the eccentric-grooved specimen is calculated for the area of metal at the base of the groove.

Table V.—Failure of Eccentric-Grooved Specimens in Solutions of Chemically Pure Sodium Hydroxide.[a]

25 g. NaOH per 100 g. H_2O. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Failure, hr.	No Failure, days
No. 23.6	67 400	13	
No. 23.7	68 600	4	
No. 23.8	65 000	8	
No. 23.9	65 000	6	
No. 23.10	62 000	18	
No. 23.11	62 000	80[b]	
No. 23.14	59 000	6	
No. 23.15	59 000	19	
No. 24.7	55 000	37	
No. 24.18	55 000	36	
No. 24.19	50 000	21	
No. 24.20	50 000		12
No. 24.24	45 000		10

---

[a] Na_2SiO_3 present is 0.02 per cent of NaOH concentration.
[b] Specimen stopped and reloaded after 50 hr.

Table II shows the result of eight tests in the embrittlement bombs to determine the tensile load to produce failure of the concentric-ground specimens in short-time tests. These tests were carried out at 250 C. with progressive increase in the load over a period of about 2 hr. until failure occurred. The breaking load of 82,200 lb. per sq. in. is somewhat higher than shown in Table I for the standard solid specimens at 250 C. and this is believed to be due to differences in shape of the specimen and rate of loading. These results were checked on tubular specimens in the tension testing machine, and good agreement was obtained. It will be noticed in this table that the addition of sodium hydroxide did not lower the breaking load as determined by these short-time tests.

With these values as a guide three test specimens, as shown in Table III, were loaded to 75,000 lb. per sq. in. in water and in 25 g. of sodium hydrox ide per 100 g. of water for periods up to 43 days without producing failure. This load is approximately 7000 lb. per sq. in. below the short-time breaking strength. From these results, it was concluded that this concentration of sodium hydroxide did not produce any appreciable reduction in the ability of the concentric-ground specimen to carry a tensile load as compared with the load it could carry in water.

Exactly the same tests were then repeated with the eccentric-grooved specimens. The results of the tests to determine the short-time breaking strength in the bombs at 250 C. are shown in Table IV. The average value for the breaking strength in water is approximately 71,000 lb. per sq. in., and the last two tests in the table show that this specimen will carry a load of 65,000 lb. per sq. in. in water for at least 15 days.

In 25 g. of sodium hydroxide per 100 g. of water, the eccentric-grooved specimens as shown in Table V failed at loads as low as 50,000 lb. per sq. in. but did not fail at 45,000 lb. per sq. in. These results were quite different from those obtained with the concentric-ground specimen and would indicate that chemically pure sodium hydroxide materially reduces the load-carrying ability of the eccentric grooved specimen. Similar results were obtained with a specimen concentrically rather than eccentrically grooved, so it may be concluded that chemically pure sodium hydroxide may have a definite effect in lowering the load-carrying ability of the steel wherever stress concentrations exist. The magnitude of the effect will probably depend to a considerable extent on the degree of stress concentration.

The first five results in Table VI show that concentrations of chemically pure sodium hydroxide materially below 25 g. per 100 g. of water will not cause the specimen to fail even at a load of 62,000 lb. per sq. in. At 45,000 lb. per sq. in., 25 g. of sodium hydroxide per 100 g. of water will not cause failure, but 50 g. will. At 40,000 lb. per sq. in. neither 50 nor 75 g. per 100 g. of water will cause failure.

It will be noticed from Tables V and VI that there was 0.02 per cent sodium silicate present as impurity in the chemically pure sodium hydroxide (Baker's). In a subsequent section of this paper it will be shown that the

Table VI.—Effect of Load and Concentration on the Failure of Eccentric-Grooved Specimens in Chemically Pure Sodium Hydroxide.

Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq in.	NaOH Concentration, g. per 100 g. H_2O[a]	Failure, hr.	No Failure, days
No. 24.22	65 000	10	..	12
No. 24.21	65 000	10	..	12
No. 23.18	62 000	10	..	10
No. 23.16	62 000	10	..	10
No. 23.20	62 000	18	..	10
No. 23.10	62 000	25	18	..
No. 23.11	62 000	25	80[b]	..
No. 23.17	62 000	50	12	..
No. 23.19	62 000	50	0.3	..
No. 24.19	50 000	25	21	..
No. 24.20	50 000	25	..	12
No. 24.24	45 000	25	..	10
No. 24.29	45 000	50	12	..
No. 24.30	45 000	50	14	..
No. 25.4	40 000	50	..	11
No. 25.6	40 000	75	..	10

---

[a] Na_2SiO_3 present is 0.02 per cent of NaOH concentration.

[b] Specimen stopped and reloaded after 50 hr.

Table VII.—Failure of Eccentric-Grooved Specimens in Sodium Hydroxide Solutions Low in Sodium Silicate.

25 g. NaOH per 100 g. H_2O. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Type of NaOH	Actual Concentration of Na_2SiO_3, g. per 100 g. H_2O	Failure, hr.
No. 23.14	59 000	c.p.[a]	0.005	6
No. 23.15	59 000	c.p.	0.005	19
No. 13.8	60 000	Low silica[b]	0.0013	21
No. 24.7	55 000	c.p.	0.005	37
No. 24.18	55 000	c.p.	0.005	36
No. 13.7	55 000	Low silica	0.0013	10

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[a] Baker's chemically pure sodium hydroxide containing 0.02 per cent Na_2SiO_3.

[b] Merck's sodium hydroxide prepared from sodium metal, containing 0.005 per cent Na_2SiO_3.

Table VIII.—Effect of Solid Sodium Sulfate on the Failure of Eccentric-Grooved Specimens in Chemically Pure Sodium Hydroxide.

All specimens covered with loose crystals of sodium sulfate. Temperature 250 C.

Specimen	Average Applied Stress, lb per sq. in.	NaOH Concentration, g. per 100 g. H_2O[a]	Failure, hr.	No Failure, days
No 24.2	62 000	25	14	..
No. 24.3	62 000	25	13	..
No. 24.25	62 000	25	13	..
No. 24.27	55 000	25	102	.
No. 24.28	55 000	25	79	..
No. 25.7	45 000	50	..	11
No. 25.8	45 000	50	..	10

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[a] Na_2SiO_3 present is 0.02 per cent of NaOH concentration
Failure time in the absence of sodium sulfate is shown in Table V.

addition of a comparatively small amount of sodium silicate has a very marked effect on the load-carrying ability of the specimens, and it is possible that even this low percentage may play some part in the failure. Two tests were run using sodium hydroxide prepared from sodium metal (Merck's) which was especially low in silica. The results shown in Table VII indicate breaking times in good agreement with those obtained with the regular chemically pure material so that it does not seem as if the low concentration of sodium silicate in these solutions produces an effect measurable by this type of testing. The low-silica sodium hydroxide was found, by colorimetric analysis, to contain about one-fourth of the sodium silicate in chemically pure sodium hydroxide.

Two tests have been run on eccentric grooved specimens using sodium carbonate and sodium phosphate instead of chemically pure sodium hydroxide, the first test with 20 g. of sodium carbonate per 100 g. of water and the second with 9 g. of trisodium phosphate per 100 g. of water. Both specimens were loaded to 65,000 lb. per sq. in., and failure did not result in 10 days, indicating that these salts at the concentrations stated do not have the same effect as sodium hydroxide.

Table IX.—Effect of Potassium Chromate on the Failure of Eccentric-Grooved Specimens in Chemically Pure Sodium Hydroxide.

25 g. NaOH per 100 g. H_2O.[a] Temperature 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	K_2CrO_4 Concentration, g. per 100 g. H_2O	Failure, hr.
No. 26.11	60 000	15	149
No. 26.14	60 000	62	625

[a] Na_2SiO_3 present is 0.02 per cent of NaOH concentration.
Failure time in the absence of potassium chromate is shown in Table V.

The tests reported in Table VIII indicate the effect of solid sodium sulfate on the failure of the eccentric-grooved specimens in chemically pure sodium hydroxide. With 25 g. of sodium hydroxide per 100 g. of water the sodium sulfate did not stop the failure of the specimens, although at 55,000 lb. per sq. in. the time required for failure is slightly longer than was found for similar tests when no sulfate was present. At 50 g. of sodium hydroxide per 100 g. of water the addition of the sodium sulfate did prevent failure for 11 days under conditions that had previously resulted in failure within 14 hr. Examination of these specimens after the run showed that they were well-covered with a thick layer of solid sodium sulfate, and it was thought that this might have formed an actual barrier between the solution and the steel. Attempts to form a solid layer on the specimens at higher loads in 25 g. of sodium hydroxide per 100 g. of water were never successful, and it was not found possible to prevent failure with sodium sulfate in this concentration of sodium hydroxide.

Table IX shows that potassium chromate in high enough concentration will greatly delay the failure of the eccentric-grooved specimens in chemically pure sodium hydroxide. It will be shown later that chromate tends to disappear during the course of the run, and it is believed necessary to start with a high concentration to insure the presence of some chromate throughout the entire test. It will be noticed that even with a fairly high concentration, in the last test failure was not completely prevented, but the specimen did carry the load for about 26 days.

Summarizing, the study of the effect of chemically pure sodium hydroxide on steel indicates: First, chemically pure sodium hydroxide does not cause a measurable reduction in the tensile load which a steel specimen will carry if the stress is uniformly distributed. Second, chemically pure sodium hydroxide reduces the load which the eccentric-grooved specimen will carry from 65,000 lb. per sq. in. in water to below 45,000 lb. per sq. in. in concentrations of sodium hydroxide up to 50 g. per 100 g. of water. Third, this effect of the chemically pure sodium hydroxide does not appear to be due to the low concentration of sodium silicate which is present as impurity. Fourth, addition of solid sodium sulfate did not prevent failure of the eccentric-grooved specimens in 25 g. of sodium hydroxide per 100 g. of water, but did prevent failure for periods up to 11 days at lower loads in 50 g. per 100 g. of water. Fifth, sufficiently high concentrations of potassium chromate prevented failure for periods up to 26 days.

TABLE X.—ROOM TEMPERATURE PROPERTIES OF BOILER FLANGE STEEL CHARGED WITH HYDROGEN BY ELECTROLYSIS.

Specimen has reduced section 0.4 in. square by 2.5 in. long.
Tensile strength of boiler-flange steel in normal condition 58,000 lb. per sq. in.; elongation 40 per cent.

Duration of Electrolysis Before Loading, hr.	Current for Electrolysis, amp.	Load, lb. per sq. in.	Time Under Load, hr.	Tensile Strength, lb. per sq. in.	Reduction in Tensile Strength from Normal, lb. per sq. in.	Elongation, per cent	Reduction in Elongation from Normal, per cent	Solution
6.......	1	50 000-55 000	0.5	Not broken	..	..	..	0.35 *N* H_2SO_4
18.......	0.25–1	50 000	27	55 000	3000	13	27	0.35 *N* H_2SO_4
20.......	0.75	50 000	24	56 000	2000	18	22	40 g. NaOH and 0.2 g. Na_2SiO_3 per 100 g. H_2O

Comparison of the Action of Chemically Pure Sodium Hydroxide With the Action of Electrolytic Hydrogen on Steel Stressed in Tension

The chemical reactions that occur when iron is placed in contact with water ordinarily result in the formation of an oxide of iron and the liberation of hydrogen from the water. Good evidence has been advanced to show that high concentrations of sodium hydroxide increase the rate of this reaction, especially at high temperatures (**1**).[2] The presence of hydrogen from these reactions has led to the belief that this gas may be an important factor in the embrittlement of steel and consequently in the failure of boiler plates. The tests reported in Table X were made to compare the physical properties and the external and microscopic appearance of specimens, fractured in tension while they were charged with hydrogen, with those of specimens fractured in tension while in contact with chemically pure sodium hydroxide solutions.

Before discussing the comparative effect, certain differences in experimental procedure should be noted. In the first place the hydrogen embrittlement tests were all run at room temperature while the sodium hydroxide tests were all run at 250 C. Second, a solid specimen 0.4 in. square was used for the hydrogen tests while the concentric-ground tubular specimen was used for the sodium hydroxide tests. Third, due to the fact that in the hydrogen tests the nascent hydrogen was released by electrolysis from an acid or alkaline solution it is probable that these specimens contained a much higher concentration of hydrogen than those broken at 250 C. in the sodium hydroxide. The effect of these differences on the results is not accurately known, but it will be seen, nevertheless, that a rough comparison can be made.

Hydrogen was evolved at the surface of the specimen for several hours before the load was applied, and the evolution of the gas was continued throughout the entire test. The tensile load was applied to the hydrogen-embrittled specimens by means of a beam and lever type Olsen testing machine. Due to the gradual elongation of the metal the load would drop slightly and it was usually raised to the original value every 8 or 10 hr.

The results shown in Table X indicate that the tensile strength is reduced only very slightly by the hydrogen, but the elongation is cut to about one-third. Comparing the results for these tests with those shown in Table II it is found that the sodium hydroxide causes no marked decrease in tensile strength compared to that obtained in water, but the elongation in the more concentrated sodium hydroxide solutions is markedly less than that obtained in water.

Figure 4 compares the external appearance of specimens broken in water and sodium hydroxide at 250 C., with the appearance of the specimen which contained hydrogen. The first two photographs were taken of the test specimens reported in Table II. Since the solid specimen embrittled by hydrogen had a much longer reduced section than the tubular specimens, the cracks appeared over a larger section, and consequently the photograph had to be taken at a lower magnification. The photographs show con-

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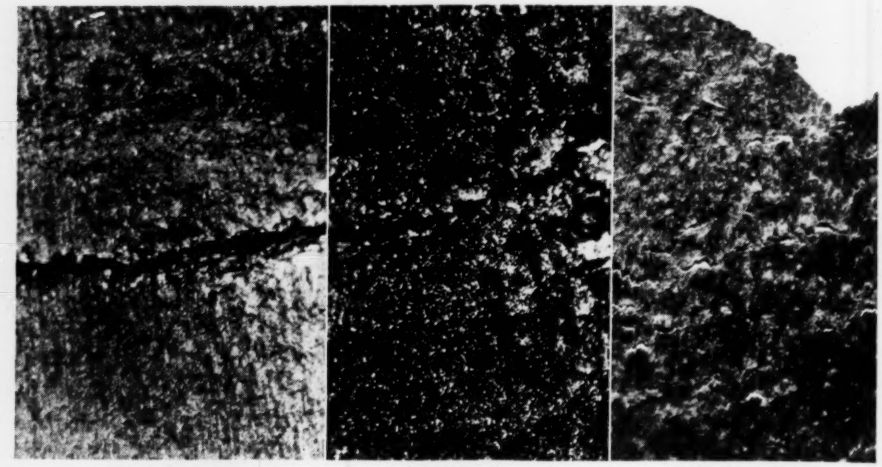
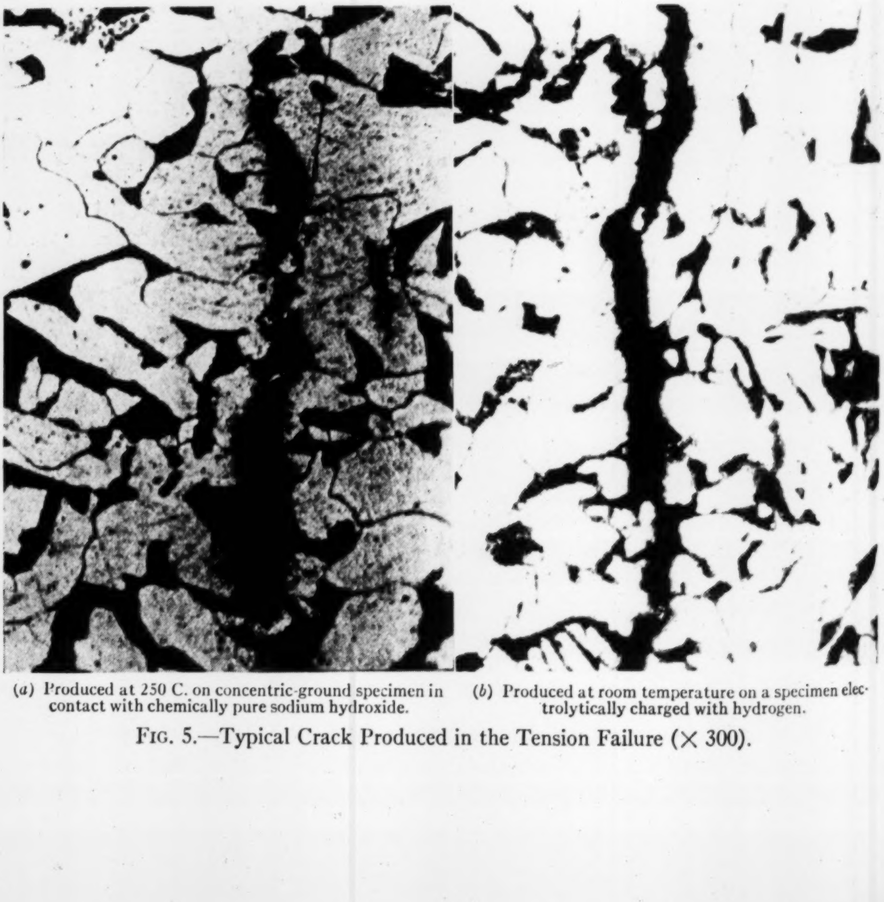
[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 750.



(*a*) Broken in water. (*b*) Broken in chemically pure sodium hydroxide. (*c*) Charged with hydrogen.

FIG. 4.—Comparison of the Fractures Produced in Water and Chemically Pure Sodium Hydroxide Solutions at 250 C. with the Fractures Produced at Room Temperature in a Specimen Electrolytically Charged with Hydrogen.



(*a*) Produced at 250 C. on concentric-ground specimen in contact with chemically pure sodium hydroxide. (*b*) Produced at room temperature on a specimen electrolytically charged with hydrogen.

FIG. 5.—Typical Crack Produced in the Tension Failure (× 300).

siderable similarity between the external appearance of the hydrogen-embrittled steel and the external appearance of steel fractured in chemically pure sodium hydroxide solution. These specimens are markedly different from the one in water. The small blisters on the hydrogen embrittled specimen did not appear on the one in sodium hydroxide (2, 3).

Figure 5 shows photomicrographs of typical cracks produced in the two specimens. The sections were prepared for examination by polishing the surface exposed to the action of the solution or gas. The cracks are rather wide and not very deep. It is difficult to distinguish their exact position with respect to the grain boundaries, but careful study has led the authors to believe that they go around and through grains in both cases. It is possible that longer exposure to the hydrogen might produce a somewhat different type of cracking, which would be more nearly intercrystalline. A few specimens of both types have been sectioned at a depth of 2 to 4 mm. below the surface, but no cracks of appreciable length were found.

Williams and Homerberg, in some tests conducted with electrolytic hydrogen passing through a specimen for a period of about 30 days, show photographs of cracks which appear to be intercrystalline. They do not state that they found the presence of the hydrogen to cause a reduction in tensile strength (2). Some tests carried out with electrolytic hydrogen for the Permutit Co. (4) under slightly different conditions did not yield intercrystalline cracks. Pfeil reports a tendency toward intercrystalline cracks at room temperature, but transcrystalline cracks at 40 to 50 C. (5). He reports only a very slight reduction in tensile strength due to the action of the hydrogen.

The similarities resulting from the tension fracture of specimens in chemically pure sodium hydroxide and specimens charged with hydrogen are: (1) Similar external appearance of fracture which is different from normal tension failure, (2) both specimens show reduced elongation, (3) there is a similar type of microscopic cracks, (4) tensile strength is not materially lowered in either case, and (5) hydrogen may be present in both cases.

The agreement between these factors indicates some justification for the belief that hydrogen is an important factor in the effect of chemically pure sodium hydroxide on steel. Further tests should be run to determine the presence of hydrogen in the steel and to see whether electrolytic hydrogen has a marked effect on notched specimens.

Particular attention should be called to the fact that this discussion has not dealt with the effect of sodium silicate - sodium hydroxide solutions. These solutions produce results which are not so readily compared with those produced by hydrogen embrittlement.

ACTION OF SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS ON THE ECCENTRIC-GROOVED SPECIMENS

It has been found that the tensile load a steel specimen can sustain at an elevated temperature in contact with a sodium hydroxide solution is markedly lowered by the addition of a small amount of sodium silicate to the solution. The effect of sodium silicate - sodium hydroxide solutions on the concentric-ground specimens was discussed in a previous paper (12) and it will not be necessary to repeat these data. In Table V it was shown that 25 g. of chemically pure sodium hydroxide would not cause failure of

TABLE XI.—LOAD REQUIRED FOR THE FAILURE OF ECCENTRIC-GROOVED SPECIMENS IN SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS.

25 g. NaOH per 100 g. H_2O. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Na_2SiO_3 Concentration, g. per 100 g. H_2O	Failure, hr.	No Failure, days
No. 15.5	40 000	1.6	73	
No. 15.9	40 000	0.40	40	
No. 15.14	40 000	0.16	21	
No. 15.20	35 000	0.16	33	
No. 15.27	30 000	0.16	93	
No. 15.24	25 000	0.16	..	10[a]
No. 15.25	25 000	0.40	190	
No. 15.17	20 000	0.32	78	
No. 15.16	15 000	0.32	..	11

[a] Microscopic examination showed embrittlement cracks.

TABLE XII.—CONCENTRATION OF SODIUM HYDROXIDE REQUIRED FOR THE FAILURE OF ECCENTRIC-GROOVED SPECIMENS IN SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS.

Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Concentration, g. per 100 g. H_2O		Failure, hr.	No Failure, days
		NaOH	Na_2SiO_3		
No. 24.22	65 000	10[a]		..	12
No. 24.21	65 000	10[a]		..	12
No. 15.12	60 000	10	0.75	54	..
No. 15.15	60 000	10	0.30	50	..
No. 15.11	60 000	7.5	0.75	..	14
No. 15.13	60 000	7.5	0.30	166	..
No. 15.7	60 000	7.5	0.15	120	..
No. 15.18	60 000	5.0	1.9[b]	..	10
No. 15.23	60 000	5.0	0.75[b]	..	10
No. 15.28	60 000	5.0	0.30	..	10
No. 16.4	60 000	5.0	0.15	..	18
No. 13.20	50 000	18	0.15	12	..
No. 13.22	40 000	18	0.15	64	..
No. 14.19	50 000	10[c]	0.30	..	10
No. 14.6	50 000	10	0.30	..	10

[a] NaOH contains 0.02 per cent Na_2SiO_3.
[b] Specimen covered with a very tightly adhering scale.
[c] Solution also contained 10 g. NaCl per 100 g. water.

the eccentric-grooved specimen at loads below 50,000 lb. per sq. in. Table XI shows that the addition of sodium silicate results in failure at loads as low as 20,000 lb. per sq. in. At 15,000 lb. load failure was not encountered.

Table XII shows the effect of concentrations of sodium hydroxide below 25 g. per 100 g. of water. The first two results in the table indicate that the specimen will stand a load of 65,000 lb. per sq. in. for at least 12 days in sodium hydroxide alone. With the addition of sodium silicate, failure resulted at 60,000 lb. per sq. in. at concentrations as low as 7.5 g. per 100 g. of water. At a load of 50,000 lb. per sq. in. 10 g. would not cause failure, but 18 g. caused failure in a relatively short time. From these results it may be concluded that the lowest concentration of sodium hydroxide which will cause failure at any given temperature is related to the highest local stress the specimen suffers. This use of the word stress does not indicate the average applied stress, as used in design calculations, but instead the stress existing at any given point in the metal. This is perhaps better expressed as an intergranular stress and it may have a value several times that of the average applied stress (6).

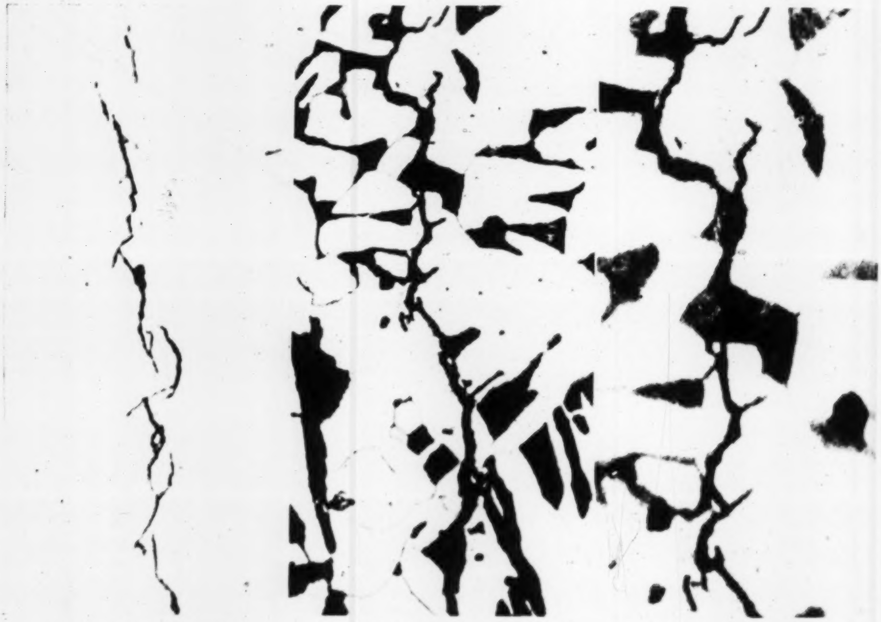
Table XIII.—Effect of High Concentrations of Sodium Hydroxide on the Failure of Eccentric-Grooved Specimens in Sodium Silicate - Sodium Hydroxide Solutions.

Temperature, 250° C.

Specimen	Average Applied Stress, lb. per sq. in.	Concentration, g. per 100 g. H_2O		Failure, hr.	No Failure, days
		NaOH	Na_2SiO_3		
No. 15.17	20 000	25	0.32	78	..
No. 15.27	30 000	25	0.16	93	..
No. 15.9	40 000	25	0.40	40	..
No. 15.14	40 000	25	0.16	21	..
No. 4.2	30 000	50	0.17	..	10
No. 4.8	40 000	50	0.16	..	10[a]
No. 4.12	40 000	50	0.17	34	..
No. 14.28	30 000	75	0.18	..	10[a]
No. 13.9	30 000	75	0.18	..	10
No. 4.11	40 000	75	0.18	..	10
No. 4.16	40 000	75	1.8	16	..
No. 4.17	40 000	75	1.8	9[a]	..

[a] Load applied at room temperature.

Table XIII shows the effect of high concentrations of sodium hydroxide in the presence of sodium silicate. Failure occurred at loads from 20,000 to 40,000 lb. per sq. in. in 25 g. of sodium hydroxide per 100 g. of water, but did not occur at 30,000 lb. per sq. in. in 50 g. of sodium hydroxide per 100 g. of water. At 75 g. of sodium hydroxide per 100 g. of water failure was not encountered until the sodium silicate concentration was increased to 1.8 g. per 100 g. of water. At a constant sodium silicate concentration these results would indicate that high concentrations of sodium hydroxide tend to delay failure of the specimen. Approximately the same result is reported by Straub from tests with commercial sodium hydroxide in which the concentration of sodium silicate was increased only in direct proportion to that of the sodium hydroxide (7). The last five results in Table XIII show that the relationship between the concentration of sodium silicate and sodium hydroxide may be of marked importance in the production of embrittlement.



(a) Unetched (× 300). (b) Etched in 2 per cent nitric acid (× 300). (c) Etched in 2 per cent nitric acid (× 600).

FIG. 6.—Cracks Produced by the Combined Action of a Sodium Silicate - Sodium Hydroxide Solution at 250 C.

Table XIV shows the effect of varying the concentration of sodium silicate in a solution containing an added sodium hydroxide concentration of 25 g. per 100 g. of water. Failure did not occur at 0.08 g. of sodium silicate per 100 g. of water, but did occur at all other concentrations up to 15 g. The results tend to show that failure occurs most rapidly at fairly low concentrations of sodium silicate. Small differences in the time required for failure are not in general believed to be of significance, but approximately the same result was obtained in the previous tests with concentric-ground specimens (12) which may indicate that high concentrations of sodium silicate do not favor the reaction to produce failure.

Cracks produced in the specimen by the action of sodium silicate - sodium hydroxide solutions are shown in Fig. 6, at high and low magnification. This is a section of the surface exposed to the action of the solution which has been polished for examination. The cracks show a tendency to be intercrystalline. Comparison with Fig. 5 will show that the type of crack produced in sodium silicate - sodium hydroxide solutions appears quite different from that produced by the action of chemically pure sodium hydroxide, or by electrolytic hydrogen.

Exclusive of temperature variation, which has been fixed for the purpose of this discussion, there are three variables known to be of primary importance in the embrittlement of steel: (1) sodium hydroxide concentration, (2) sodium silicate concentration, and (3) the stress existing in the steel. With these variables a great many combinations are possible and the four tables that have been presented for the action of sodium silicate - sodium hydroxide solutions cannot be interpreted as more than an outline which will serve as a guide for further testing. Other factors, such as the effect of possible accelerating agents, may also be found to be of considerable importance.

TABLE XIV.—EFFECT OF THE CONCENTRATION OF SODIUM SILICATE ON THE FAILURE OF ECCENTRIC-GROOVED SPECIMENS IN SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS.

25 g. of NaOH per 100 g. H_2O.[a] Load, 40,000 lb. per sq. in. Temperature, 250 C.

SPECIMEN	Na_2SiO_3 CONCENTRATION, G. PER 100 G. H_2O	FAILURE, HR.	NO FAILURE, DAYS
No. 16.5	0.08	..	11
No. 15.14	0.16	21	..
No. 15.9	0.4	40	..
No. 15.5	1.6	73	..
No. 15.1	7.5	83	..
No. 16.3	15	78	..

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[a] Concentration expresses amount of sodium hydroxide added to the solution exclusive of that introduced in combination as sodium silicate.

INFLUENCE OF SODIUM SULFATE, ON THE ACTION OF SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS

The experimental investigation of the influence of sodium sulfate on the failure of eccentric-grooved specimens in sodium silicate - sodium hydroxide solutions is of especial interest due to the general use of this salt in actual practice to delay or prevent the embrittling of steel. A number of tests have been run on the effect of sodium sulfate present as a solid and in solution. While the data are not so extensive as it is hoped they may ultimately become, they do present some very interesting angles regarding the protective action of this salt.

In practically all of the tests previously described in this paper, the load has been applied to the specimen after the bomb had been brought to the operating temperature of 250 C. About 2 or 3 hr. are required for the bomb to reach this temperature after it has been inserted in the air thermostat and the current turned on. Using this procedure, the first four results in Table XV do not indicate that covering the specimen with loose crystals of solid sodium sulfate has any marked effect in retarding failure. In the second test, aside from covering the test specimen with sodium sulfate the bomb was also rotated at 250 C. for several hours and transferred while hot to the embrittlement thermostat and the load applied. This step was taken to insure saturation of the solution with sodium sulfate before application of the load. This did not prevent failure. In tests No. 16.30 and 13.14, 10 g. of solid were dissolved in the solution as well as covering the specimen with solid, but failure still occurred in about the usual time.

TABLE XV.—INFLUENCE OF SOLID SODIUM SULFATE ON THE ACTION OF SODIUM SILICATE-SODIUM HYDROXIDE SOLUTIONS.

25 g. NaOH per 100 g. H_2O. All specimens covered with crystals of solid sodium sulphate. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Na_2SiO_3 Concentration, g. per 100 g. H_2O	Failure, hr.	No Failure, days	Temperature at Load Application, deg. Cent.	Remarks
No. 16.6	40 000	0.18	56	..	250	
No. 15.4	40 000	0.37	23	..	250	Rotated at 250 C. to insure saturation.
No. 16.30	30 000	0.18	80	..	250	10 g. Na_2SO_4 in solution.
No. 13.14	40 000	0.18	12	..	250	10 g. Na_2SO_4 in solution.
No. 16.2	40 000	0.18	197	..	room	
No. 16.11	30 000	0.18	160	..	room	
No. 13.13	40 000	0.18	135	..	room	
No. 15.6	40 000	0.37	216	..	room	
No. 13.12	40 000	0.18	...	10	room	10 g. Na_2SO_4 in solution.
No. 16.29	30 000	0.90	...	10	room	10 g. Na_2SO_4 in solution.
No. 13.11	40 000	1.8	...	10	room	10 g. Na_2SO_4 in solution.
No. 13.23	40 000	5.0	...	10	room	10 g. Na_2SO_4 in solution.

Since practical experience and experimental study have strongly indicated that sodium sulfate does have some influence on the embrittlement of steel, the experimental procedure was changed in one respect in an attempt to detect this effect. The specimens were covered with loose crystals of solid but instead of applying the load after the bomb had reached operating temperature it was applied at room temperature before heating was started. All four of these samples shown in the second series of tests in Table XV indicate a definite increase in the number of hours required for failure compared to the failure time in the absence of sodium sulfate. None of the specimens, however, lasted more than 10 days. In the last four tests in the table, instead of adding all of the sodium sulfate as solid, about 10 g. were dissolved in the solution before it was put into the bomb and then the loose crystals of solid were added. This insured a concentration of about

12 g. of sodium sulfate per 100 g. of water at the start of the test, which is approximately saturation at room temperature. The load was applied at room temperature, and in every case the specimen lasted for 10 days. It will also be noticed that failure was not produced even in sodium silicate concentrations as high as 5 g. per 100 g. of water.

These tests led to the belief that sodium sulfate in solution might have a definite effect in retarding failure. In the first test in Table XVI the sodium sulfate was completely dissolved at 250 C. by rotation of the bomb over night in a thermostat; it was then quickly transferred to the embrittlement thermostat and the load applied. Failure occurred in a very short time. In the next test 10 g. of sodium sulfate were dissolved in the solution before it was put into the bomb to give a concentration of approximately 16 g. per 100 g. of water, and the load was again applied at 250 C. Failure occurred in the usual time.

Table XVI.—Influence of Sodium Sulphate in Solution on the Action of Sodium Silicate - Sodium Hydroxide Solutions.

25 g. NaOH per 100 g. H_2O. Sodium sulfate dissolved in solution before introducing into bomb. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Concentration, g. per 100 g. H_2O		Failure, hr.	No Failure, days	Temperature at Load Application, deg. Cent.
		Na_2SO_4	Na_2SiO_3			
No. 15.3	40 000	29[a]	0.3	5	..	250
No. 14.8	40 000	16	0.16	36	..	250
No. 16.16	30 000	1.6	0.16	35	..	room
No. 16.27	40 000	8	0.16	45	..	room
No. 13.19	40 000	10	0.16	108	..	room
No. 13.27	40 000	12	0.16	40	..	room
No. 14.5	40 000	12	0.16	...	10	room
No. 16.19	30 000	16	0.16	...	10	room
No. 16.22	40 000	16	0.16	...	10	room
No. 14.10	40 000	16	0.16	...	10	room
No. 14.15	40 000	16	0.48	...	10	room
No. 16.25	30 000	16	0.80	68	..	room
No. 14.23	40 000	16	1.6	117	..	room

[a] Bomb rotated at 250 C. overnight to reach saturation. Small amount of excess solid present.

The next 8 tests in Table XVI were carried out by dissolving increasing concentrations of sodium sulfate up to the saturation value at room temperature, in the solution before it was poured into the bomb and applying all of the loads at room temperature. Concentrations below 12 g. per 100 g. of water did not indicate any marked protective effect. One specimen at this concentration did, however, run for 10 days, and three specimens with a concentration of 16 g. per 100 g. of water ran for 10 days. The last three results in the table show that if the concentration of sodium silicate is increased to a value of 0.8 g. per 100 g. of water or above the presence of 16 g. of sodium sulfate per 100 g. of water will not prevent failure.

These results show that sodium sulfate in solution has a very definite retarding effect on the action of the sodium silicate - sodium hydroxide solutions. Furthermore, it is necessary to have a fairly high concentration to exert a measurable influence and the beneficial effect of solid may be that it tends to insure saturation. If this is true solid sodium sulfate may prevent failure in the solutions containing high sodium silicate by raising the sodium sulfate concentration from 16 g. at room temperature to about 29 g. per 100 g. of water at 250 C.

The reason why a specimen covered with crystals of solid is not as effectively protected from the action of the solution as one which has previously had some sodium sulfate dissolved in the solution is not altogether clear. It would be expected that the presence of the crystals of solid at a surface of the specimen would insure rapid saturation of the solution. There is a possibility, however, that since the solution is not stirred diffusion through the solution film on the sodium sulfate and the film on the specimen may not be sufficiently rapid to insure saturation at all times. The results would indicate that the presence of the sodium sulfate in solution during the early part of the tests, especially during the heating period, plays a very important part in retarding the embrittlement action.

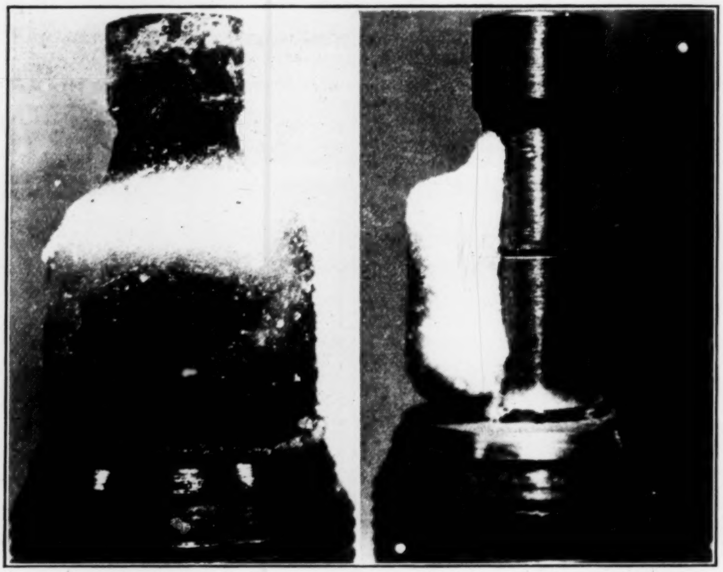

FIG. 7.—Layer of Sodium Sulfate Formed from Loose Crystals of Solid and a Cross Section of This Layer.

With solid sodium sulfate it is also possible that the specimen may be protected by a solid layer which effectively separates the solution from the steel. Figure 7 shows a specimen removed from the bomb with its surrounding layer of sodium sulfate and a cross-section of this layer, which had formed during the run from the loose crystals. The layer is very thick, and while not as dense as a calcium sulfate scale it has considerable mechanical strength and could conceivably prevent the solution from reaching the steel. Examination of the cross section in Fig. 7 will show that the sodium sulfate has actually filled the groove of the specimen. This would present a still further barrier to the penetration of the solution.

Accurate comparison of these results for the influence of sodium sulfate with those obtained by Straub in a similar study, using a commercial sodium hydroxide solution on a stressed solid specimen, cannot be made at the present time (8). Straub ran a series of tests at lower temperatures (90 to 100-lb. gage) by evaporating a dilute solution to give hydroxide concentrations around 400 or 500 g. per liter and found that failure was not prevented until the ratio of sodium sulfate to sodium hydroxide reached more than 2 to 1. At this temperature and concentration less than 7 g. of sodium sulfate per 100 g. of water will dissolve in the solution, which may be too low to exert a measurable influence. In the presence of sufficient sulfate, failure may have been prevented by a layer of sodium sulfate. Straub further ran a series of tests at 500-lb. gage in 120 to 140 g. of sodium hydroxide per liter in the presence of sodium chloride to study the action of sodium sulfate. It should be noted that the reduction of the commercial sodium hydroxide concentration to this value also reduced the sodium silicate concentration to approximately 0.08 g. per 100 g. of water which is apparently near the border line for failure or no failure. In the present study no tests have yet been run on the influence of sodium sulfate at sodium hydroxide concentrations as low as 10 or 14 g. per 100 g. of water either in the absence or presence of sodium chloride. Other tests on the action of sodium sulfate which were run by Straub indicated that solid sodium sulfate had to be in contact with the specimen to prevent failure. For these tests the sodium hydroxide concentration, the applied stress, the tendency of the solid to coat the specimen, and the exact procedure for adding the sodium sulfate to the solution are not stated. In view of these facts it does not seem desirable to make an attempt to compare the results of the investigations until further experimental work under exactly comparable conditions can be completed.

The differences secured for the influence of sodium sulfate on the action of the solutions on the eccentric-grooved specimens when they are loaded at room temperature or 250 C. have been found of considerable interest. It is known that the embrittlement reactions go slowly at temperatures below 100 C.; therefore, if the specimen is loaded and its deformation practically completed at room temperature, as the bomb heats up the sodium sulfate in solution may create a protective film that cannot be penetrated by the sodium silicate - sodium hydroxide solution. On the other hand, if the bomb is loaded at 250 C. the mechanical deformation may break any protective film that has been formed, and the embrittling action may proceed to ultimate failure more rapidly than the sodium sulfate can create a protective film. If this is the case it should be possible to break a specimen protected by sulfate after it has been loaded at room temperature by releasing the load and reapplying at 250 C. One test of this type has been carried out and failure did occur, but it is desirable to carry out more extensive tests to substantiate or disprove this theory of the action of dissolved sodium sulfate.

With solid sodium sulfate the application of the load at 250 C. may break both the oxide layer formed by the dissolved sodium sulfate and any solid sodium sulfate layer. The different loading procedures may then cause the same effect as if only dissolved sodium sulfate were present.

TABLE XVII.—INFLUENCE OF SODIUM PHOSPHATE ON THE ACTION OF SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS.

25 g. per 100 g. H_2O. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Concentration, g. per 100 g. H_2O		Failure, hr.	No Failure, days	Temperature at Load Application, deg. Cent.
		Na_3PO_4	Na_2SiO_3			
No. 16.9[a]	40 000	0.2	0.16	14	..	250
No. 16.7	40 000	1.6	0.16	16	..	250
No. 4.3	40 000	5[b]	0.80	42	..	250
No. 13.28	40 000	5[b]	0.16	162	..	250
No. 16.12	30 000	0.48	0.16	...	11	room
No. 16.17	40 000	0.48	0.16	227	..	room
No. 4.5	40 000	1.0	0.16	...	10	room
No. 4.4	40 000	5.0	0.16	...	10	room
No. 13.29	40 000	5	0.16	...	10	room
No. 14.1	30 000	0.48	0.8	47	..	room
No. 14.4	30 000	2.4	0.8	38	..	room

[a] Commercial sodium hydroxide containing 0.64 per cent Na_2SiO_3.
[b] Solution saturated and about 3 g. of excess solid present in the bomb.

The results secured in the study of the influeuce of sodium sulfate on the action of sodium silicate - sodium hydroxide solutions on steel may be summarized as follows: (1) Sodium sulfate did not retard failure when the load was applied at 250 C., (2) sodium sulfate in solution and solid sodium sulfate both retarded failure when the load was applied at room temperature, (3) a sodium sulfate solid layer may act as a mechanical barrier between the solution and the steel to produce an effect on the time required for failure.

Practically these results would indicate that sodium sulfate would be most effective in preventing the embrittlement of steel where static stresses predominated and there is little possibility of repeated stress or mechanical distortion of the metal. The influence of this salt in solution cannot be relied on to produce complete protection since there is no evidence to show that it is not the plugging of the capillary seams with solid which is the really effective preventative of the embrittlement of the steel. It may also be more difficult for a repeated stress to destroy the influence of this plugging effect than for such a stress to destroy the influence of dissolved sodium sulfate. If the primary effect is one of plugging the small openings, other dissolved solids in the boiler water may be of as much value as the sodium sulfate. If sodium sulfate is relied on to prevent embrittlement it seems desirable to keep as high a concentration in solution in the boiler water as compatible with satisfactory operation since in that case the influence of the dissolved sodium sulfate is supplemented by plugging of capillary spaces. This should create conditions least favorable to embrittlement.

It is apparent from this discussion of the action of sodium sulfate that the experiments which have been run only furnish an outline for the action of this salt under certain specific conditions. It is highly desirable that tests be run over a much wider range of conditions.

TABLE XVIII.—INFLUENCE OF SODIUM CHROMATE, SODIUM NITRATE AND POTASSIUM PERMANGANATE ON THE ACTION OF SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS.

25 g. NaOH per 100 g. H_2O. Average applied stress, 40,000 lb. per sq. in. All loads applied at 250 C. Temperature, 250 C.

Specimen	Concentration, g. per 100 g. H_2O		Failure, hr.	No Failure, days
	Added Salt	Na_2SiO_3		
No. 16.13	Na_2CrO_4 0.16	0.16	121	..
No. 16.8	Na_2CrO_4 1.6[a]	0.16	...	10
No. 14.12	Na_2CrO_4 1.6[b]	0.16	...	10
No. 14.13	Na_2CrO_4 1.6	0.80	...	10
No. 16.10	$NaNO_3$ 1.6[c]	0.16	...	11
No. 16.15	$KMnO_4$ 1.6[d]	0.16	...	10

[a] Chromium almost disappeared from solution during the test.
[b] Solution also contained 10 g. NaCl per 100 g. H_2O.
[c] Nitrate almost disappeared from solution during test.
[d] Trace of manganate left at end of test.

INFLUENCE OF SOME COMPOUNDS OTHER THAN SODIUM SULFATE ON THE ACTION OF SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS

The first four samples in Table XVII show the effect of trisodium phosphate on specimens loaded at 250 C. None of the tests, with the possible exception of No. 13.28, indicated a measurable influence. The slight increase in time of failure for this specimen is believed to be due to the formation of a phosphate layer from the excess solid which was present. The next five specimens, loaded at room temperature, all indicated a definite beneficial effect of the sodium phosphate in solution. The last two specimens show that higher concentrations of sodium silicate have an adverse effect on the influence of the phosphate in retarding failure. It will be noticed from this table that sodium phosphate exerts a definite influence in increasing the time to failure at much lower concentrations than sodium sulfate.

The first four results in Table XVIII show that sodium chromate will greatly increase the time required for failure, even when the load is applied to the specimen at 250 C. Furthermore, 1.6 g. of sodium chromate per 100 g. of water prevented failure for more than 10 days with sodium silicate concentrations as high as 0.8 g. per 100 g. of water.

Sodium nitrate and potassium permanganate were also effective in preventing failure of the specimens when the load was applied at 250 C. The nitrate almost disappeared from the solution during the test, and the strong odor of ammonia from the bomb lead to the belief that a considerable proportion had been converted to this gas through reduction by hydrogen and iron. Some manganate was present in the solution at the end of run No. 16.15 but there was also a considerable amount of precipitate in the solution which was probably MnO_2. Chromium in all forms practically disappeared from solution during run No. 16.8. This was the only run in which disappearance was almost complete; in the other runs, some residual chromate was found in the solution at the end of the test.

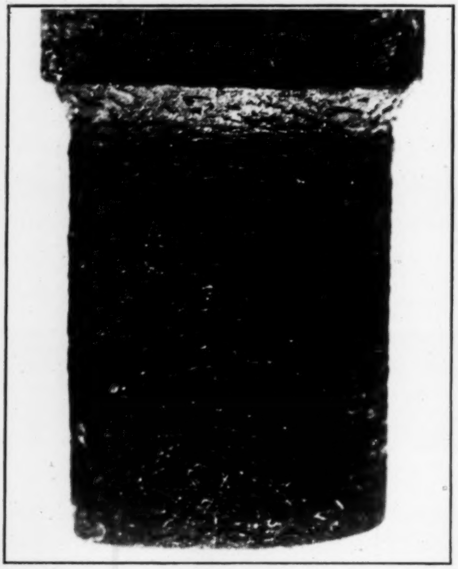

FIG. 8.—Film Formed by the Action of a Sodium Chromate Solution Which Was Flaked Off by the Action of a Fresh Sodium Silicate - Sodium Hydroxide Solution.

The disappearance of the chromium during test No. 16.8 led to the belief that the specimen might have been covered with an iron chromium film, and the stability of this film was tested by reloading the specimen at

250 C. and 40,000 lb. per sq. in. in a solution containing 25 g. of sodium hydroxide and 0.16 g. of sodium silicate per 100 g. of water. Failure occurred in 48 hr. When the broken specimen was removed from the bomb the film was found to be flaked off as shown in Fig. 8. The small flakes could easily be removed from the surface, and qualitative analysis indicated that they contained a considerable quantity of chromium.

Berl has also found that chromate largely disappeared from a solution in contact with iron filings at 310 C. (1). The chromium appeared on the surface of the iron filings and he found furthermore that the presence of the chromate did not retard the oxidation of the iron powder but it did retard the evolution of hydrogen gas. This would indicate that if an iron chromium film is formed it is not a very good barrier to keep the solution away from the steel and that one of the prime effects of the chromate is the oxidation of the hydrogen gas evolved in the reaction between iron and water.

TABLE XIX.—INFLUENCE OF SODIUM SULFITE ON THE ACTION OF SODIUM SILICATE - SODIUM HYDROXIDE SOLUTIONS.

25 g. of NaOH per 100 g. H_2O. 0.16 g. Na_2SiO_3 per 100 g. H_2O. Temperature, 250 C.

Specimen	Average Applied Stress, lb. per sq. in.	Na_2SO_3 Concentration, g. per 100 g. H_2O	Failure, hr.	No Failure, days	Temperature at Load Application, deg. Cent.
No. 16.14	40 000	3.2	16	. .	250
No. 13.24	40 000	16	29	. .	250
No. 4.7	40 000	1.0	80	. .	room
No. 13.25	40 000	3.2	. .	10	room
No. 14.20	40 000	8	. .	10	room
No. 14.14	30 000	16	. .	10	room

Straub has run tests with phosphate, chromate and nitrate, which appear to agree in at least a qualitative manner with the results just shown (9). When Straub ran his tests neither the effect of sodium silicate nor the differences resulting from loading the specimen at room temperature or some higher temperature was clearly understood and it has been found possible to extend the data concerning the influence of these salts on the embrittlement reactions by variation in these factors.

Table XIX shows the effect of sodium sulfite on the action of sodium silicate - sodium hydroxide solutions. When the load is applied at 250 C., failure occurs in the usual time, even with a sodium sulfite concentration of 16 g. per 100 g. of water. If the specimen is loaded at room temperature, however, 3.2 g. of sodium sulfite will prevent failure for at least 10 days.

The results show that sodium phosphate, sodium chromate, sodium nitrate, sodium permanganate, and sodium sulfite all have a very definite influence, under certain conditions, in retarding the action of the sodium silicate - sodium hydroxide solutions. With the exception of sodium phosphate and perhaps sodium sulfite it does not seem desirable to introduce any of the other compounds into the boiler water until further studies have been carried out concerning their decomposition products.

OXIDIZING PROPERTIES OF SODIUM SULFITE WITH RESPECT TO HYDROGEN OR IRON

All of the salts shown in this report to have a retarding influence on the action of the sodium silicate - sodium hydroxide solutions have contained oxygen. Furthermore the compounds which have powerful oxidizing properties at room temperature were more effective than other compounds.

TABLE XX.—REDUCTION OF SODIUM SULFITE BY HYDROGEN AND IRON.

Temperature, 250 C.

Test	Solution Charged					Solution from Bombs			
	Total Water, g.	Total Steel Saw-ings, g.	Dissolved Solids, g. per 100 g. H_2O			Concentration, g. per 100 g. H_2O[b]			Sulfite Reduced as Calculated from Iodine Titration, g. Na_2SO_3 per 100 g. H_2O[c]
			Na_2SO_3	Na_2SO_4	NaOH[a]	Na_2SO_3	Na_2SO_4	NaOH[a]	
No. 885....	154	25	1.77	0.32	2.5	sampler 1.56	0.35[e]	2.5	0.19
						bomb 1.54	0.35	2.5	0.18
No. 889....	150	60	0.57[d]	0.08[e]	0.55	sampler 0.01	0.08[e]	0.56	0.43
						bomb 0.02	0.08	0.56	0.38
No. 891....	150	60	0.47[d]	0.05[e]	0.42	sampler 0.01	0.05[e]	0.43	0.39
						bomb 0.01	0.05	0.44	0.33
No. 893....	150	60	0.4[f]	0.09[e]	0.34	sampler 0.01	0.09[e]	0.35	0.29
						bomb 0.02	0.09	0.36	0.25
No. 894....	165	none	0.35[f]	....	0.5[g]	sampler 0.19	0.10[e]	0.43	0.002
						bomb 0.22	0.10	0.46	0.008
No. 895....	165	none	0.35[f]	....	0.5[g]	sampler 0.19	0.11[e]	0.49	0.004
						bomb 0.21	0.11	0.52	0.006

[a] Determined by titration from phenolphthalein to methyl red.

[b] Concentrations expressed on this basis are effected to a small extent by the disappearance of water due to the reaction with iron.

[c] This calculation is based on the assumption that all of the sodium sulfite which was reduced went to sodium sulfide and the results are expressed in terms of Na_2SO_3 per 100 g. of H_2O to make possible ready comparison with the solution charged into the bomb.

[d] Value from total sulfur in solution. This includes sodium sulfite and sodium sulfate.

[e] Maximum sodium sulfate that could be present as determined from bomb analysis.

[f] Weighed into bomb as dry powder

[g] Approximate concentration added to bomb.

The tests run with sodium sulfite were carried out with the expectation that this salt, since it was believed to act as a reducing agent at boiler temperatures, might actually accelerate the embrittling action of the solutions. This was not true and these tests have led to some interesting results concerning the properties of sodium sulfite at 250 C. Iron at high temperature is such a powerful reducing agent that it can break down water to release hydrogen. The tests in Table XX show that either iron or the released hydrogen can reduce sodium sulfite at 250 C.

These tests were run by charging a solubility bomb made of steel with a dilute solution containing sodium hydroxide, sodium sulfite and saw cuttings from the boiler flange steel, rotating the bomb at 250 C. for 48 hr. or more, and then sampling the solution. Both the sample, remaining solution in the bomb and the original solution were analyzed for hydroxide, sulfate, sulfite, and the combined sum of the thiosulfate and sulfide. In the titration for reducing agents other than sulfite, the removal of the effect of this ion was accompolished by the addition of formaldehyde to the solution (10). The method of titration was checked on the solution originally charged into the bomb with results of high accuracy.

The first test in Table XX shows only a small amount of reduction of the sodium sulfite, but the next three tests with lower concentrations of sodium sulfite and sodium hydroxide as well as the addition of more steel sawings gave almost 100 per cent reduction of the sulfite. This means that the sulfite is an oxidizing agent with respect to hydrogen and iron. Since under the existing conditions the iron must necessarily be a more powerful reducing agent than hydrogen in order that it may break down water, it would be anticipated that the decomposition of the sulfite might occur to a considerable extent at the surface of the iron. It is well known that at room temperature in acid solution zinc will reduce sulfite and with this reaction in mind the reduction of sulfite by iron at 250 C. might be anticipated. The last two results in the table show that very little of the sulfite was reduced in the absence of the steel sawings. The small amount of reduction encountered was probably produced by the steel walls of the bomb. These tests also indicate that the sodium sulfite does not suffer auto-reduction by the following reaction:

TABLE XXI.—EFFECT OF IRON AND HYDROGEN ON SODIUM SULFATE.

Temperature, 250 C.

Titration with 0.1 *N* iodine on aliquots of these samples required less than 0.05 ml. in every case. This is within experimental error and indicates that no reducing agent was present in the samples.

Slight increases in concentration which are shown for both sulfate and hydroxide are probably due to the loss in water resulting from the reaction with iron.

Test	Solution Charged				Solution from Bombs, g. per 100 g. H_2O	
	Total Water, g.	Total Steel Sawings, g.	Dissolved, g. per 100 g. H_2O			
			Na_2SO_4	NaOH	Na_2SO_4	NaOH
No. 888	141	15	1.68	0.93	sampler 1.73	1.11
					bomb 1.72	1.05
No. 883	141	15	1.68	0.93	sampler 1.71	1.12
					bomb 1.71	1.01
No. 886	150	40	0.56	0.65	sampler 0.58	0.67
					bomb 0.58	0.66
No. 887	150	40	0.56	0.65	sampler 0.59	0.67
					bomb 0.57	0.66

$$4\,Na_2SO_3 = Na_2S + 3Na_2SO_4$$

Berl thought this reaction could occur but it was not found in the present tests (11).

Further tests are shown in Table XXI to determine whether sodium sulfate is reduced by iron. No reduction could be detected by this experimental method. These tests do, however, furnish conclusive proof that there was nothing in the steel sawings that could release any compound to react with the iodine in the titration. Berl also carried out some tests on the reduction of sulfate but none was found (11).

These results may explain the influence of sodium sulfite on the embrittlement reaction. It is a more powerful oxidizing agent with respect to iron than sulfate and will produce a measurable influence at lower concentrations. It probably is not so powerful an oxidizing agent as permanganate or nitrate and therefore is not so effective in preventing failure as these salts.

This oxidizing property of the sodium sulfite may throw a new light on the mechanism of the prevention of corrosion by this salt in waters containing low concentrations of oxygen. It is well known that at room temperature salts which have oxidizing properties will often produce a passive film that will greatly retard corrosion by a given solution. Similarly the oxidation of the iron by the sodium sulfite to produce a less permeable or passive oxide layer on the iron may be more important than its removal of low concentrations of oxygen. This possibility should receive further consideration in any study of the action of sodium sulfite and it may well change the emphasis from the maintenance of a reducing salt in the boiler water to prevent corrosion, to the maintenance of a salt having the correct oxidizing properties with respect to the iron at the temperature in question. The practical results obtained with sodium sulfite indicate that it may have approximately the correct properties.

CONCLUSIONS

The following tentative conclusions may be drawn from the work discussed in this progress report.

1. Chemically pure sodium hydroxide has not been found to reduce the uniformly distributed tensile load a specimen will carry as compared to the load it will sustain in water alone.

2. Chemically pure sodium hydroxide causes a marked reduction in the load-carrying ability of a specimen subject to a stress concentration such as caused by a groove. Experimental tests indicate that this effect is probably not due to the low concentration of sodium silicate present in the chemically pure sodium hydroxide.

3. It has been shown that chemically pure sodium hydroxide produces results on specimens stressed in tension at 250 C. that are similar in a number of ways to those obtained on tension specimens electrolytically charged with hydrogen at room temperature. The introduction of sodium silicate to the sodium hydroxide solution apparently destroys a number of the points of similarity.

4. The introduction of small amounts of sodium silicate into sodium hydroxide solutions greatly lowers the load required to produce failure at 250 C. in specimens tested in tension alone, and in specimens suffering a concentration of stress.

5. The lowest concentration of sodium hydroxide required to cause failure of eccentric-grooved specimens in sodium silicate - sodium hydroxide solutions depends to a considerable extent on the load applied to the specimen.

6. Sodium sulfate retarded the failure of the eccentric-grooved specimens in sodium silicate - sodium hydroxide solutions only when the load was applied at room temperature. High concentrations of sodium sulfate in solution, and a solid layer of sodium sulfate may both play a part in the influence of this salt. At the present time it appears desirable to maintain as high a concentration of sulfate in the water as compatible with satisfactory boiler operation, since this should create conditions least favorable to embrittlement. The protection offered by sodium sulfate may not be satisfactory where the steel is subjected to repeated stress.

7. Trisodium phosphate, sodium chromate, sodium nitrate, potassium permanganate, and sodium sulfite exert a definite influence on the action of the sodium silicate - sodium hydroxide solutions under certain conditions. The more powerful oxidizing agents appear to be the most effective, but due to their decomposition further experimental work must be done before it will be possible to recommend their introduction into a boiler water. Sodium chromate appears to form a film of iron and chromium on the surface of the specimen.

8. Sodium sulfite is reduced by iron and hydrogen at 250 C. This reduction may explain its influence on the embrittlement reaction as well as offer a new view of its effect in the prevention of corrosion.

Practically all of the work described in this paper is in good agreement with the results reported in the rather extensive literature on the embrittlement of boiler steel. Investigation of the effect of the sodium silicate has made it possible to extend and correlate our knowledge and to resolve existing conflicts in the literature. At the present time a complete understanding of the chemical reactions involved in embrittlement has not been secured, but recent intensive investigation has brought a number of new and vital factors to light. As a result of this work it is now possible to offer at least reasonable explanations as to why boilers operating under conditions which are generally satisfactory have at times encountered difficulties; or why boilers operating under unsatisfactory water conditions have not had trouble. Furthermore, it is not difficult to understand why embrittlement has frequently been reported for locomotive operation under conditions which have been found satisfactory in stationary service. The many experimental investigations now being carried out should not only yield a great deal of information concerning the embrittlement of steel but should also further our knowledge concerning corrosion or other reactions which may occur in boiler waters at high temperature.

Acknowledgments:

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DISCUSSION

MR. E. B. POWELL[1] (*presented in written form*).—No one can have followed the work carried forward under Mr. Partridge and now under Mr. Schroeder at New Brunswick, of which this paper presents the latest progress report, without being impressed by its precision, the open mindedness and the clarity of thought with which every phase of the research has been planned, and the speed with which these results have been secured. While acquaintance with the authors may preclude surprise from the quality of results, the extent and scope of the attainments now recorded have certainly exceeded the expectations of any of those even remotely associated with the project. I trust that I may not be out of order in including the expression of confidence that, as the true merit and value of this work comes to be realized, the power industry as a whole will not fail to accord generously the support which is essential for its completion.

To my mind the two most outstanding of the attainments of the research at New Brunswick so far have been the identification of silica as of importance in the embrittling of boiler plate and the experimental support given the theory of film inhibition of the embrittling action.

As has been pointed out by the authors, the discovery of the importance of silica in the phenomenon under consideration has gone a long way toward explanation of the inconsistencies of earlier work as reported by different investigators. As also suggested by the authors, there are doubtless other not uncommon constituents of boiler water whose presence in particular concentration relations tends to accelerate the embrittling attack.

The difference in results secured under conditions of load application at room temperature and after heating of the specimen to 250 C. points rather strongly to protective value secured through a precipitated film. The protective film, it would seem from the evidence of the present research, must be a product of precipitation as only by applying the load at room temperature and then thereafter maintaining relatively stable surface dimensions of the specimen was the most effective inhibiting secured. The evidence of the stability of sodium sulfate in the presence of iron would seem to give further support by indicating that the protective film is not, or at least need not be, an oxide.

In contrast with conditions in the steam boiler, there is no progressive concentration from incoming solids taking place in the solution in the test bomb after the attainment of the operating temperature. Conceivably, in the case of the steam boiler, in the capillary seams in which the critical concentrations are attained there may under some circumstances be conditions which simulate those in the bomb in which concentration of the inhibiting salt does not proceed continuously and, in consequence, a break in the protective film as might be caused by mechanical strain is not promptly repaired, and the embrittling action is therefore not inhibited.

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Perhaps I should have said that the outstanding accomplishment of the research so far has been the revelation of the almost total absence of tenable reasoning back of our prescription for avoidance of boiler plate embrittlement. The only really substantial basis of this prescription is that it has, in stationary equipment at least, appeared to work. Without a reasoned theory, however, it is not very satisfying to base one's prescription for the protection of boilers operating at present-day high pressures merely upon the experiences of the relatively lower pressure installations, which alone have sufficient breadth in background of experience in anywise to serve as a guide. When it is considered that the trend today is toward the adoption of higher and higher pressures in the boiler supplying steam for ultimate use in processes from which there is no condensate return, calling for chemically treated water as the bulk of the boiler feed, the importance of a complete and thoroughgoing understanding of the boiler plate embrittlement phenomenon and the requirements for its prevention can hardly be overestimated.

MR. R. T. SHEEN[2] (*by letter*).—There is one point that might be considered in planning a continuation of this work so as to simulate as nearly as possible conditions as existing in operating boilers. One could not help but be impressed with the importance of reactions taking place at the interface bounded by the steel and the solution, this being most evident in a study of the effects of oxidizing agents. Previous research into the cause and mechanism of the deposition of scale has shown that chemical equilibrium is materially affected at an interface under conditions of evaporation at that interface *versus* static conditions. There can be little question but that some evaporation must take place in the lap or seam of a boiler, as otherwise it would be impossible to obtain concentrations that are found present that cause failure to occur, and in any event, it may be assumed that there is a certain equilibrium between the vapor and the liquid phase at that point.

Such conditions could be reproduced in these tests by inserting between the push rod and the test specimen a heater and furnishing these specimens with just sufficient heat to cause a small amount of evaporation from the walls of the test specimen and possibly inserting in the top of the bomb a reflux condenser so that concentrations will at all times be practically constant under a given set of conditions. The heat input to the heater could, of course, be carefully controlled so as to obtain a minimum temperature differential to the test specimen and at the same time data obtained in this fashion will perhaps more nearly simulate conditions that would be obtained in actual operating boilers. It is the writer's opinion that results obtained in this manner will differ considerably from results obtained up to the present time under the static conditions.

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[2] Technical Director, W. H. & L. D. Betz, Philadelphia, Pa.

The effect of a number of reagents other than sodium sulfate on the action of sodium silicate and sodium hydroxide solutions has been studied and up to the present time, the reagents studied have been confined entirely to the inorganic group. There are doubtless a number of those interested in this work who would like to see the results obtained with the effect of various organic materials in water included in the study such as the starches or tannins as protective types of colloid and laminaria digitata or laminaria stenophyllia as typical types of reactive colloid. With the increasing importance of work in this field of organics, the question will doubtless be asked as to the effect of these organics on the action of sodium silicate - sodium hydroxide solutions and the work which undoubtedly is intended to be fully comprehensive, should include studies of these materials to be complete.

MESSRS. W. C. SCHROEDER, A. A. BERK AND EVERETT P. PARTRIDGE (*authors' closure by letter*).—The authors wish to thank Mr. Powell and Mr. Sheen not only for the constructive criticism which they have offered, but also for the careful and detailed attention they have given this involved subject. It is to be noted that the discussions have indicated the importance of the same point, for example, the action of colloids, or the importance of surface effects.

Considerable time has elapsed since the presentation of this paper and it has been possible to carry out further investigation on a number of points. Evaporation of the solution at the surface of the specimen has not been found to give a measurable difference in the tension tests as compared to the embrittling action caused by a purely static solution. Further intensive investigation has been carried on on the effect of various solutions on the surface of steel. It has been found that very small changes in the composition of the solution produce radical changes in the surface conditions. It is hoped that this work will be reported in detail in the near future.

Investigation has also been started on the effect of a number of organic compounds, some of which are colloidal. A few of these compounds have been found to cause a very marked increase in the resistance of the steel to the action of the solutions.

At the present time sufficient data have been accumulated to make possible the formulation of a definite theory for the chemical and physical effects which enter into the production of embrittlement. This formulation is attempting to give due consideration to electrolytic, colloidal and surface effects. In the final form it should be of great aid in selecting and determining the effectiveness of possible protective compounds. It is hoped that this work can be finished well within the period of time allowed for the investigation.

THE USE OF SOLUBILITY DATA TO CONTROL THE DEPOSITION OF SODIUM SULFATE OR ITS COMPLEX SALTS IN BOILER WATERS[1]

By W. C. Schroeder, A. A. Berk, and Everett P. Partridge

Synopsis

The constituents of a boiler water are believed to exert an important effect upon the cracking of boiler steel. For example sodium silicate and sodium hydroxide tend to produce embrittlement and sodium sulfate tends to inhibit this effect.

The results of an extended investigation of the solubility of sodium sulfate from approximately 300 to 660 F. (150 to 350 C.) in water and in various complex solutions representative of concentrated boiler waters have been used to develop the curves in this paper which express the conditions under which sodium sulfate will be deposited during the evaporation of a boiler water of a given initial composition. If further investigation shows that sodium sulfate either in solution or as solid is necessary to prevent embrittlement, these curves will define the conditions which can or should be maintained.

At the present time the curves cannot be used to control the composition of boiler waters for the prevention of embrittlement unless a number of assumptions are made. Further testing now being carried out should eliminate the necessity for these assumptions and furnish data which will allow direct application in practice.

The results of an extensive investigation of the solubility of sodium sulfate from approximately 300 to 660 F. (150 to 350 C.) in water and in various complex solutions[2] have been used to develop the curves in this paper which express the conditions under which sodium sulfate or its complex salts will be deposited during the evaporation of a dilute solution of a given initial composition. It must be understood that this paper simply presents solubility data in units which make possible direct correlation between dilute and concentrated solutions.

At the present time the curves shown *cannot* be used for the calculation of ratios for the protection of steel from embrittlement. Further data are, however, being collected which it is believed will make it possible to calculate the desired protective ratios. The curves in this paper will then lead to uniform values for these ratios, as well as eliminate the neces-

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[1] Published by permission of the Director, U. S. Bureau of Mines.

This paper has been prepared in connection with an investigation conducted under a cooperative agreement between the Joint Research Committee on Boiler Feed Water Studies and the U. S. Bureau of Mines. The general investigation is supervised by a subcommittee of which J. H. Walker is chairman, and is carried out at the Nonmetallic Minerals Experiment Station of the Bureau of Mines, New Brunswick, N. J.

[2] W. C. Schroeder, A. A. Berk, and Everett P. Partridge, "The Solubility of Sodium Sulfate in Boiler Water Salines as Related to the Prevention of Embrittlement," Progress Report No. 3, presented at the A.S.M.E. meeting, December, 1934.

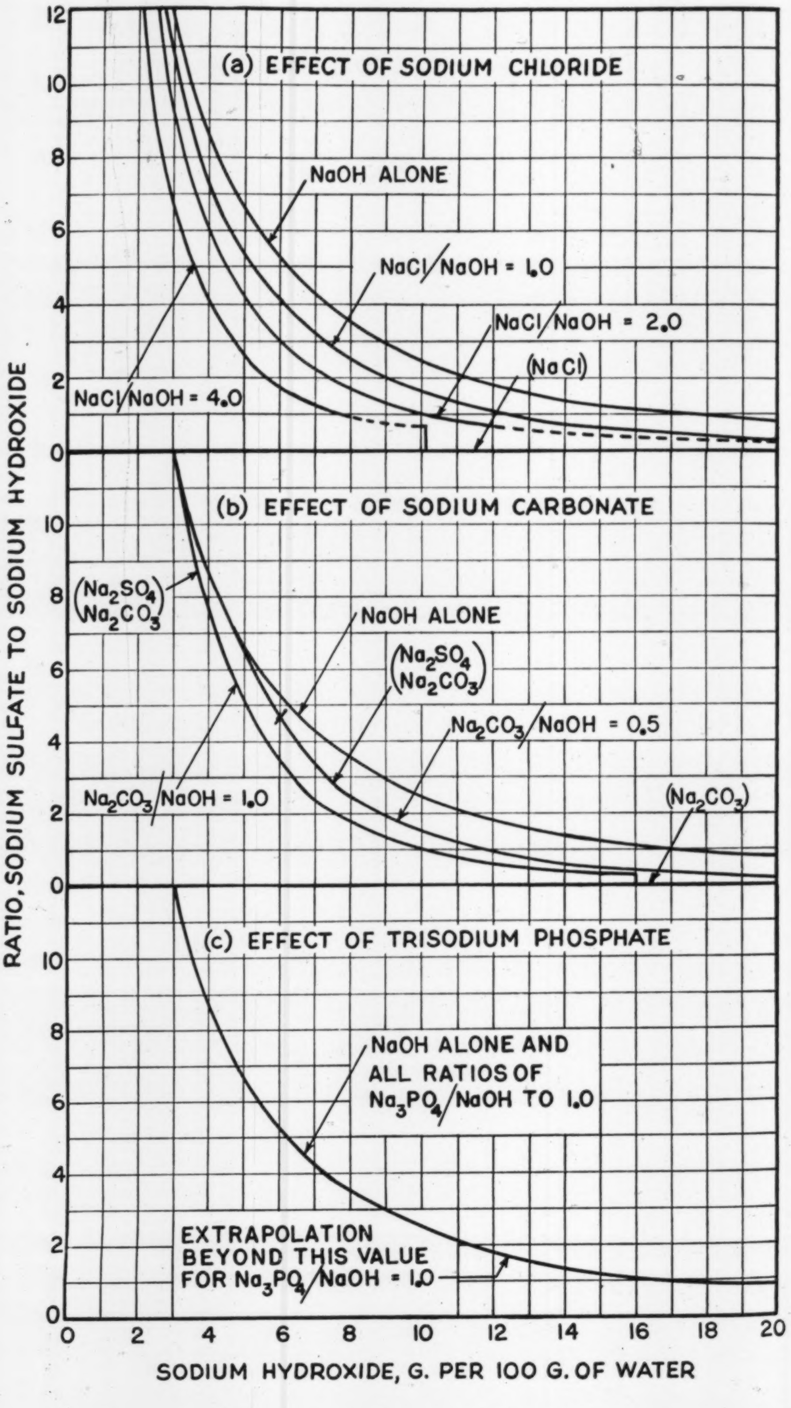

FIG. 1—Solid Deposition Curves, 300 F. (150 C.) 54-lb. gage.

sity of going through complicated calculations to obtain the results from the solubility data. They will be of value in defining the conditions which can or should be maintained in the boiler water with respect to the desirable concentrations of sodium sulfate regardless of whether this salt is found to produce protection by its presence in solution or as solid.

SOLID DEPOSITION CURVES

The solubility data used to calculate the curves shown in Figs. 1 to 5 have been discussed in detail in a progress report,[2] and are being published in a series of papers; the first two have already appeared, in the *Journal* of the American Chemical Society.[3,4]

Since the effect of sodium hydroxide on boiler steel is not believed to be due to the concentrations present in the body of the boiler water but rather to the concentrated solution resulting from evaporation or chemical reaction in the capillary spaces, it is simplest to express the amount of a given salt present as a ratio to the amount of sodium hydroxide. This procedure follows the customary method used in chemical engineering calculations, of selecting a constituent which goes through the system unchanged and referring all calculations to this constituent. In the present case sodium hydroxide should be satisfactory and it is then unnecessary to consider the number of concentrations due to evaporation as a variable.

In all the figures it will be found that the ordinate is expressed as a ratio of sodium sulfate to sodium hydroxide, and where other salts are present their concentrations are expressed in similar ratios.

The abscissa in all cases represents grams of sodium hydroxide per 100 g. of water. *This value is not related to the sodium hydroxide concentration in the actual boiler water but is determined solely as the concentration of sodium hydroxide which must be attained to cause the deposition of a solid phase starting with some ratio of sodium sulfate to sodium hydroxide as shown on the ordinate.*

Three graphs are given for each temperature; the first deals with a simple solution containing sodium sulfate and sodium hydroxide and the effect of addition of sodium chloride; the second deals with the corresponding effect of sodium carbonate on the simple solution; and the third with the corresponding effect of trisodium phosphate. This means that the boiler water has been considered as a solution containing primarily only sodium sulfate and sodium hydroxide, the solubility relations of which are modified by the presence of other sodium salts such as the chloride, the carbonate

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[3] W. C. Schroeder, Alton Gabriel, and Everett P. Partridge, "Solubility Equilibria of Sodium Sulfate at Temperatures of 150 to 350 C. I. Effect of Sodium Hydroxide and Sodium Chloride," *Journal*, Am. Chemical Soc., Vol. 57, pp. 1539–1546 (1935).

[4] W. C. Schroeder, A. A. Berk and Alton Gabriel, "Solubility Equilibria of Sodium Sulfate at Temperatures of 150 to 350 C. II. Effect of Sodium Hydroxide and Sodium Carbonate," *Journal*, Am. Chemical Soc., Vol. 58, pp. 843–849 (1936).

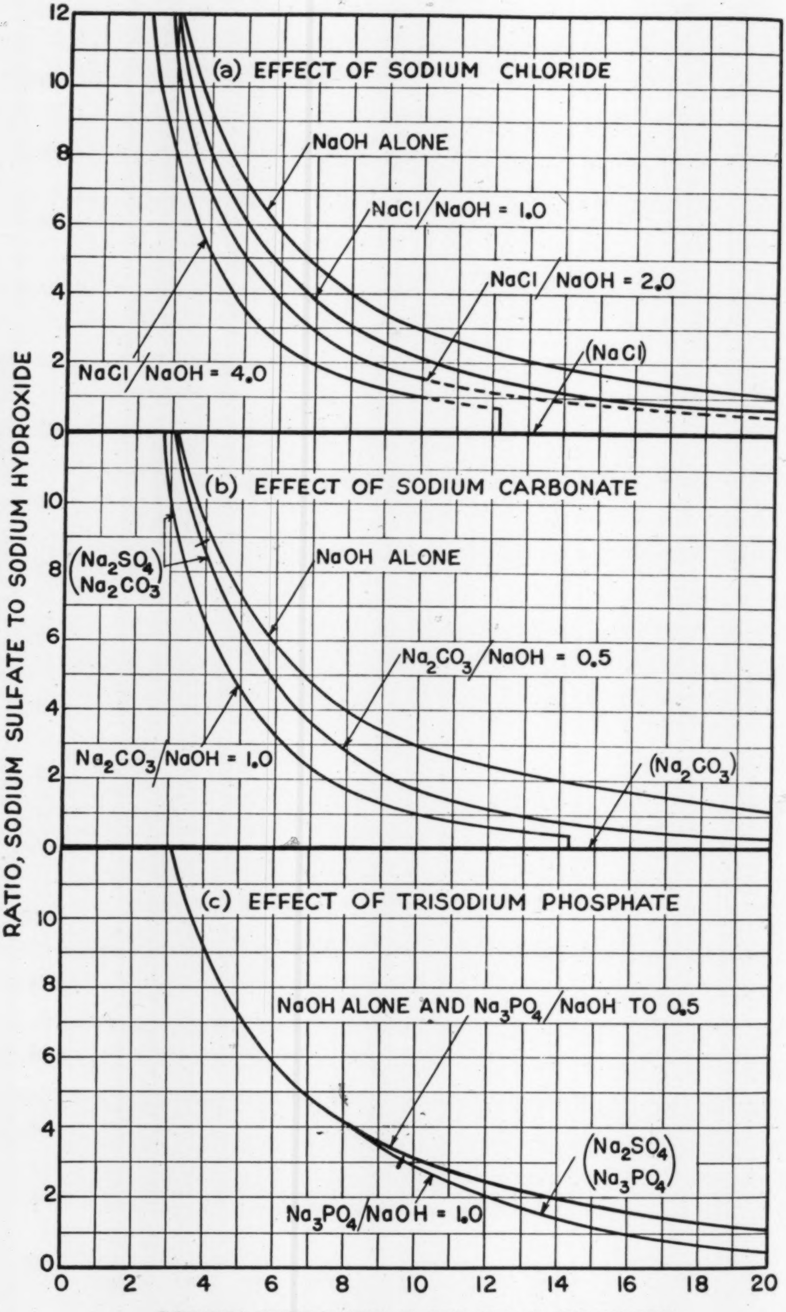

FIG. 2.—Solid Deposition Curves, 390 F. (200 C.) 210-lb. gage.

and the phosphate. Sets of three graphs have been plotted for 300, 390, 480, 570 and 660 F. (150, 200, 250, 300 and 350 C.).

The curves have all been plotted in terms of sodium hydroxide rather than alkalinity expressed as sodium carbonate in order to make them both simple and precise. For solutions containing hydroxide and carbonate or hydroxide and phosphate, the use of such a term as alkalinity as sodium carbonate would have involved two salts and would result in difficulties both in plotting and using the solid deposition curves.

The solubility data were sufficiently comprehensive so that extrapolation was unnecessary except in a few cases. Such extensions beyond the values obtained by direct determination are indicated in the figures by dotted lines.

Interpolation was necessary at 390 and 570 F. (200 and 300 C.) for the solutions containing either phosphate or carbonate. At 390 F. (200 C.) the resulting solid deposition curves are probably as accurate as those in which the actual solubility data were used. At 570 F. (300 C.) some error due to interpolation may appear but this is probably too small to be of practical significance. The effect of the concentration of sodium hydroxide in the systems containing two additional chemicals was estimated by linear interpolation.

Sodium sulfate is not, of course, the only sodium salt which may separate out of a boiler water during its concentration. For example, if the solution contains enough sodium carbonate a double salt containing sodium carbonate and sodium sulfate may be deposited. Complex solid phases encountered in the case of the phosphate are even more likely to separate out due to the higher stability of phosphate in solution at high temperatures and to the fact that these complex salts may be encountered at low phosphate concentrations. If the solid which deposits is not sodium sulfate this is indicated on the curve by enclosing in parenthesis the salt or salts found in the new solid phase. For the sodium chloride and sodium phosphate curves, the appearance of a new solid phase is usually indicated by a discontinuity. Deposition of new solid phases does not always cause a material break, especially for those phases containing sodium carbonate, and the approximate position is then indicated by a short line across the solid deposition curve. The solid composition is not indicated, however, since it depends on the dissolved salts in the solution and furthermore the composition of some of the phosphate solid phases has not been definitely established.

No direct evidence has been advanced to show that the deposition of the double salts or of sodium chloride, sodium carbonate or sodium phosphate will have the same effect as the deposition of sodium sulfate is thought to have. It is only possible to assume that this is the case until further data are secured.

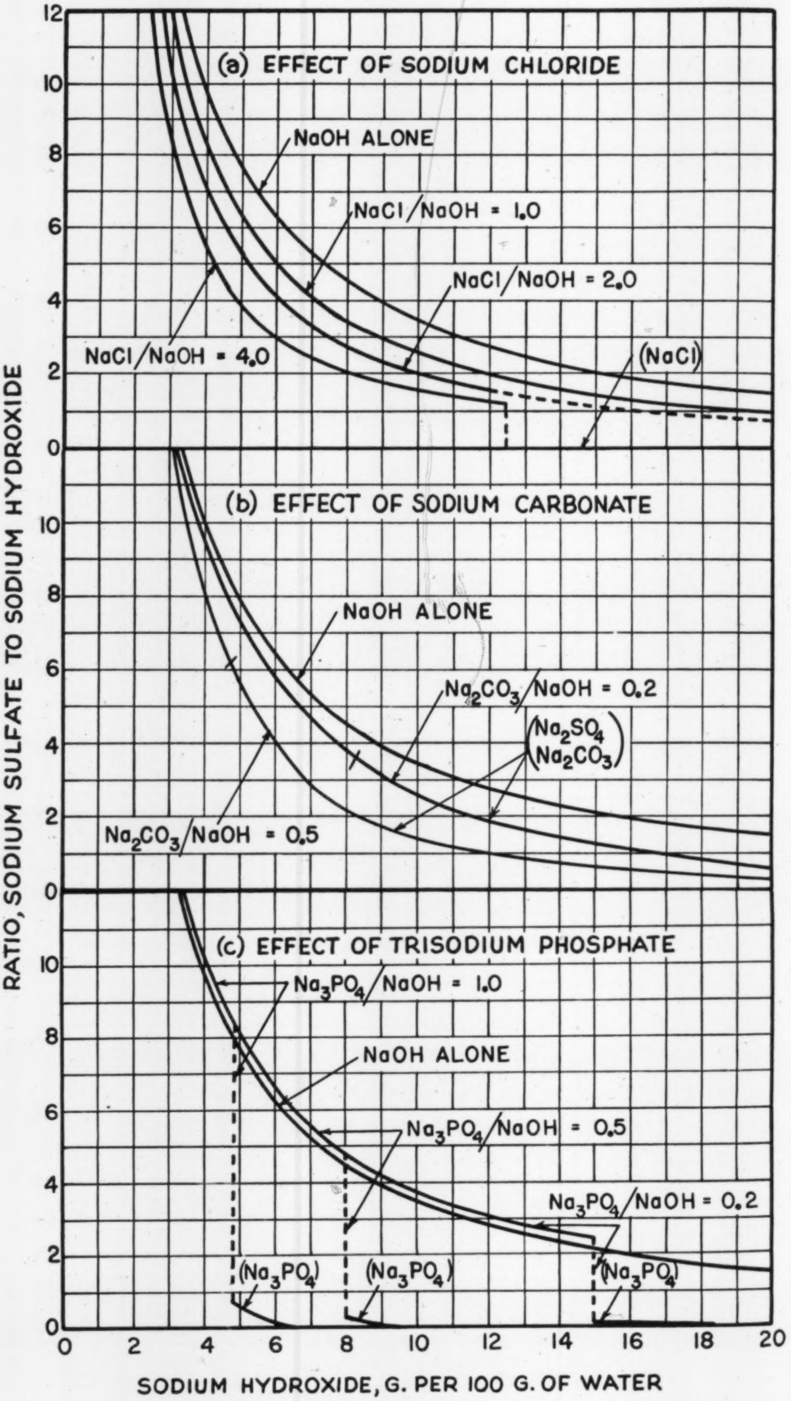

FIG. 3.—Solid Deposition Curves, 480 F. (250 C.) 561-lb. gage.

In cases where the ratio of sodium chloride, carbonate or phosphate to sodium hydroxide is sufficiently high, these simple salts may separate out as such, without the deposition of any sodium sulfate. This means that the $Na_2SO_4/NaOH$ ratio necessary for the deposition of solid may fall to zero. Several instances of this will be found in the figures.

On the phosphate curves at the higher temperatures, the solubility data show first a sodium sulfate branch, then an almost vertical series of double salt branches and finally a sodium phosphate branch. In plotting the solid deposition curves, the sodium sulfate branch has been followed as long as it existed and then the double salt branch existing between the sodium sulfate and sodium phosphate phases has been treated as if it were exactly vertical. This results in a discontinuity in the solid deposition curves and is indicated in the figures by a vertical dotted line. This same procedure has also been used to indicate discontinuities for the other salts if any were encountered.

Sodium chloride, as shown by the curves, will be found to deposit at the lower temperatures when the $NaCl/NaOH$ ratio is high. The value taken as the point for deposition was obtained from the water solubility curve since no data are available for the solubility of sodium chloride in sodium hydroxide solutions. The effect of sodium hydroxide or sodium sulfate would probably be to lower this solubility so that the $Na_2SO_4/NaOH$ ratio given may be slightly higher than is absolutely necessary.

The sodium phosphate curve at 300 F. (150 C.) for sodium phosphate to sodium hydroxide ratios near unity has been extrapolated for sodium hydroxide concentrations above 12 g. per 100 g. of water. This has been done on the assumption that no double salt is formed. If such a double salt does form, the $Na_2SO_4/NaOH$ ratio should be slightly lower than shown. The sodium phosphate curve at 660 F. (350 C.) is continuous for $Na_3PO_4/NaOH$ ratios from zero to slightly below 0.1. In Fig. 5 (*c*) it will be found that the curve is indicated as continuous for ratios up to 0.15. This indicates a higher $Na_2SO_4/NaOH$ ratio than is necessary for the ratios between 0.1 and 0.15.

EXPLANATION FOR USE OF CURVES

The solid deposition curves may be used in two ways: first, with a given boiler analysis to determine the concentration of sodium hydroxide which will be attained before solid deposition starts; or second, to determine the ratio of sodium sulfate to sodium hydroxide which will cause solid deposition before any certain, selected, sodium hydroxide concentration is attained.

In the first method the procedure is as follows:

1. From the analysis of the boiler water calculate the $NaOH$, Na_2SO_4, $NaCl$, Na_2CO_3, and Na_3PO_4 concentrations. These must be calculated in terms of the chemical compounds indicated and not in terms of ions. Since

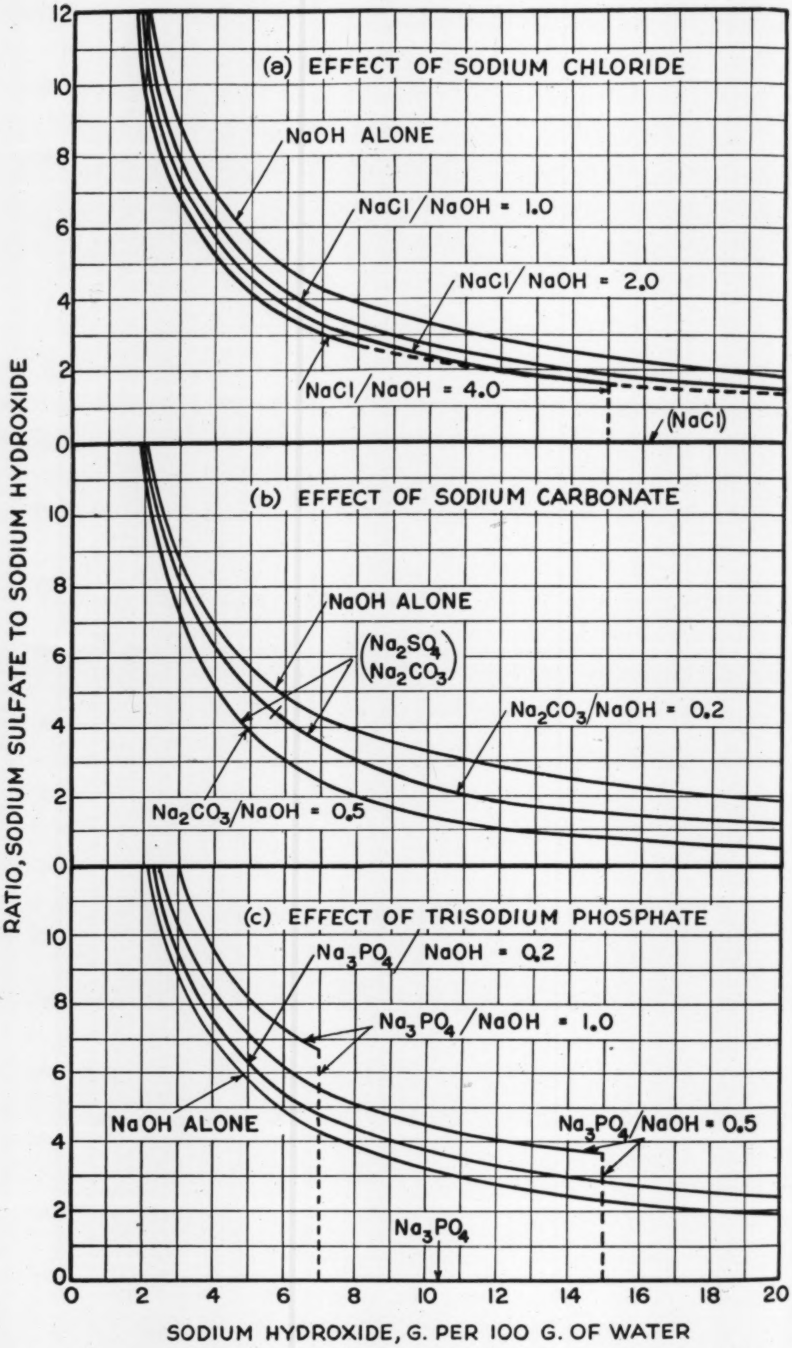

FIG. 4.—Solid Deposition Curves, 570 F. (300 C.) 1282-lb. gage.

Figs. 1 to 5 are based on weight ratios, concentrations must be expressed on a weight basis instead of in terms of chemical equivalents. It is immaterial whether the boiler water composition is calculated in terms of parts per million or grains per gallon.

2. Calculate the ratio of each salt present to the amount of sodium hydroxide.

3. Select the set of curves nearest the operating temperature or pressure of the water in the boiler and move on a horizontal line for the Na_2SO_4/NaOH ratio until a curve is intersected which is nearest the NaCl/NaOH ratio of the boiler water. The concentration of sodium hydroxide at this point where precipitation of a solid phase begins is indicated by the value on the abscissa.

If this value for the sodium hydroxide concentration is higher than is desirable before solid deposition starts, it will be necessary to increase the sodium sulfate concentration in the boiler water or to decrease the sodium hydroxide concentration.

The same reading should be made on the plots for sodium carbonate and for sodium phosphate if these salts are present in appreciable concentrations. The lowest figure obtained for the sodium hydroxide concentration may be taken as the value which will initiate precipitation of the solid phase. In general, since the addition of other sodium salts tends to decrease the solubility of the sodium sulfate in the sodium hydroxide solutions this allows a desirable slight factor of safety.

An exception exists to this use of the lowest sodium hydroxide concentration at a temperature of 570 F. (300 C.) when the Na_3PO_4/NaOH ratio is high. Here it will be found that the presence of the phosphate increases the concentration of sodium hydroxide and it will be well to deduct this increase in a linear manner from the decrease caused by the presence of other salts before reading the sodium hydroxide concentration which causes solid deposition.

If no plot is presented for a pressure near the operating pressure, linear interpolation may be used on the values obtained at the pressures just below and just above the operating pressure. Linear interpolation may also be used to estimate the position of curves for NaCl/NaOH, Na_2CO_3/NaOH or Na_3PO_4/NaOH ratios between those shown. Such interpolation will be accurate in the majority of cases since the ratio curves are quite regular.

If it is desired to assume a certain sodium hydroxide concentration and to calculate the Na_2SO_4/NaOH ratio which will deposit solid at this concentration of sodium hydroxide, the steps are much the same as before. The assumed concentration on the abscissa is followed along a vertical line until it intersects the correct curve for the boiler-water ratio of NaCl, Na_2CO_3 or Na_3PO_4 to NaOH and the Na_2SO_4/NaOH ratio is read on the ordinate. If the sodium hydroxide concentration of the boiler water is

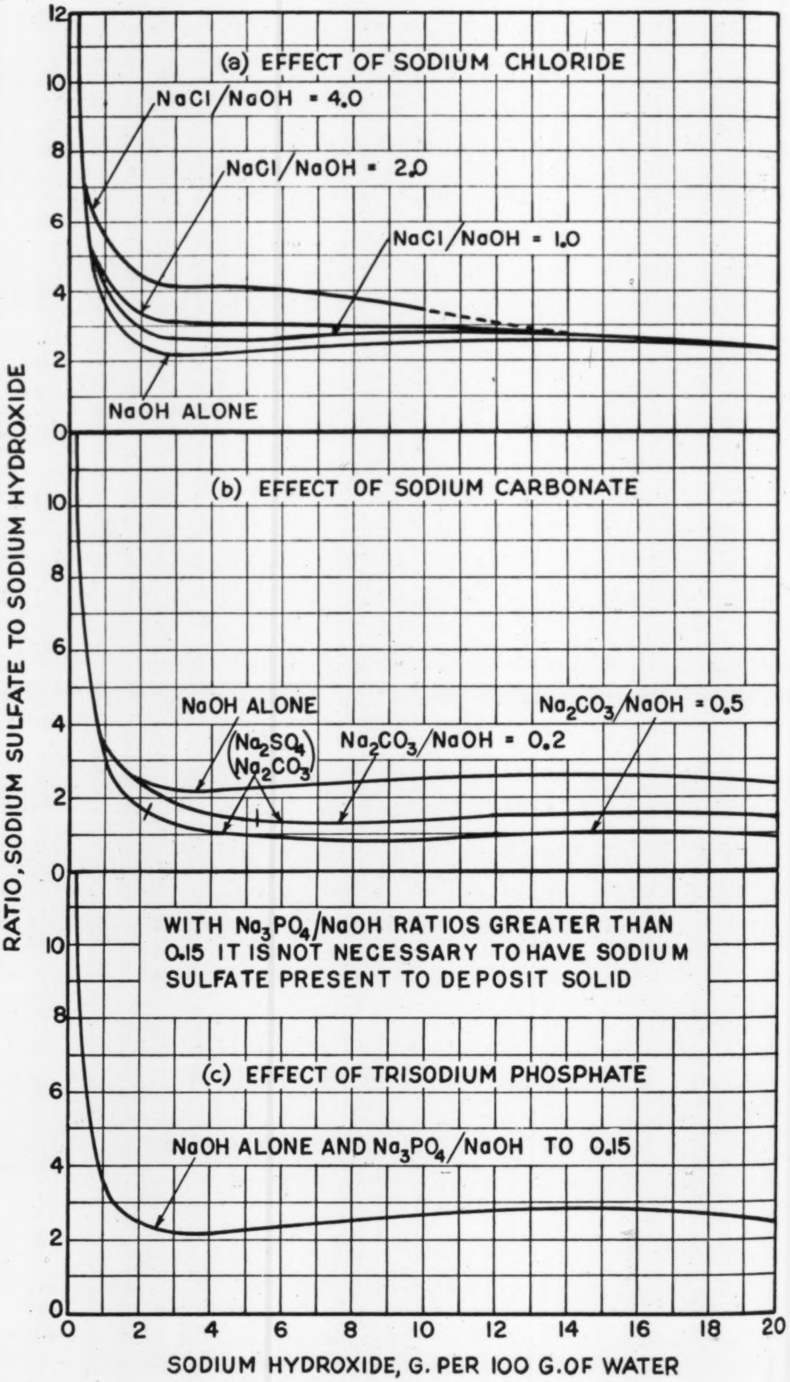

FIG. 5.—Solid Deposition Curves, 660 F. (350 C.) 2386-lb. gage.

multiplied by this value, the sodium sulfate which should be present in the boiler water is obtained.

Examples will make this discussion somewhat clearer. Assuming the following boiler-water analysis:

Example I:

ASSUMED BOILER WATER.

Operating pressure, 200-lb. gage, 390 F. (200 C.).

ION	SALT	RATIO, SALT TO NAOH
OH (200 p.p.m.)	NaOH (471 p.p.m.)	..
SO_4 (1000 p.p.m.)	Na_2SO_4 (1480 p.p.m.)	3.14
Cl (100 p.p.m.)	NaCl (165 p.p.m.)	0.35
CO_3 (25 p.p.m.)	Na_2CO_3 (44 p.p.m.)	0.09

Figure 2(*a*) and (*b*) corresponds closely to the operating pressure. Interpolating for the NaCl/NaOH ratio in Fig. 2(*a*), the value of the sodium hydroxide concentration is 8.7 g. per 100 g. of water. For the Na_2CO_3/NaOH ratio in Fig. 2(*b*) the value is approximately 9. The lower value of 8.7 is the one at which the deposition of solid sodium sulfate will start.

Example II:

For a second example it is possible to calculate the amount of sodium sulfate necessary in a boiler water to deposit solid sodium sulfate when the water has concentrated to attain 10 g. of sodium hydroxide per 100 g. of water. The operating pressure is 500-lb. gage and the analysis is as follows:

ASSUMED BOILER WATER.

Operating pressure, 500-lb. gage, 470 F. (245 C.).

ION	SALT	RATIO, SALT TO NAOH
OH (300 p.p.m.)	NaOH (706 p.p.m.)	..
SO_4 (800 p.p.m.)	Na_2SO_4 (1184 p.p.m.)	..
CO_3 (10 p.p.m.)	Na_2CO_3 (18 p.p.m.)	0.03
PO_4 (35 p.p.m.)	Na_3PO_4 (60 p.p.m.)	0.08

The set of curves nearest this operating pressure are shown in Fig. 3(*a*), (*b*) and (*c*) at 480 F. (250 C.). From Fig. 3(*b*) and (*c*) and the ratios for Na_2CO_3/NaOH and the Na_3PO_4/NaOH it is apparent that the concentrations of these salts are too low to be of importance. At a sodium hydroxide concentration of 10 in Fig. 3(*a*), the necessary Na_2SO_4/NaOH ratio is 3.5. This means that in this boiler water there should be $3.5 \times 706 = 2470$ p.p.m. of sodium sulfate and that under the assumed conditions the 1184 p.p.m. Na_2SO_4 present will not cause solid deposition when 10 g. of sodium hydroxide per 100 g. of water is attained.

Example III:

For a third example the effect of high concentration of sodium chloride on example II will be considered. The analysis is now as follows:

ASSUMED BOILER WATER.

Operating pressure, 500-lb. gage, 470 F. (245 C.).

ION	SALT	RATIO, SALT TO NAOH
OH (300 p.p.m.)	NaOH (706 p.p.m.)	..
SO_4 (800 p.p.m.)	Na_2SO_4 (1184 p.p.m.)	..
Cl (500 p.p.m.)	NaCl (824 p.p.m.)	1.17
CO_3 (10 p.p.m.)	Na_2CO_3 (18 p.p.m.)	0.03
PO_4 (35 p.p.m.)	Na_3PO_4 (60 p.p.m.)	0.08

The effect of phosphate and carbonate are again negligible and at 10 g. of sodium hydroxide per 100 g. of water with this high concentration of sodium chloride Fig. 3(*a*) shows the necessary $Na_2SO_4/NaOH$ ratio to be 2.3. This means that in the boiler water there should be $2.3 \times 706 = 1624$ p.p.m. of sodium sulfate. The effect of the chloride has been to reduce the sulfate necessary from 2470 p.p.m. as in example II to 1624 p.p.m. The 1184 p.p.m present are still not sufficient under the assumed conditions.

It is possible to determine the concentration of sodium hydroxide in the boiler water with sufficient accuracy for use in these calculations as follows:

1. Titrate with acid to the phenolphthalein end point and multiply by the correct factor to convert the acid titer to parts per million of NaOH.

2. Multiply the known parts per million of Na_2CO_3 present by 40/106 and the known parts per million of Na_3PO_4 present by 40/164 and deduct the sum of these two values from the value calculated above (paragraph 1). If other buffer salts are not present this will give a value relatively close to the true sodium hydroxide concentration. These statements apply to boiler water containing appreciable concentrations of sodium hydroxide and relatively small concentrations of carbonate and phosphate. Where low concentrations of NaOH are present, as for boiler waters having a pH value of 10.5 or lower, it may be best to calculate the hydroxide concentrations from the pH value as suggested by McKinney.[5,6]

It is believed that this explanation and the examples will make clear the use of the graphs shown in Figs. 1 to 5. These figures quite accurately picture the necessary conditions for starting the deposition of solid. They do not, of course, give any information concerning the quantity of solid deposited per unit of water evaporated. It should be noted that they are not only based on extensive solubility data but that these data have been checked in solutions undergoing evaporation to establish the differences

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[5] D. S. McKinney, "Interpretation of Water Analysis," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, pp. 192–197 (1931).

[6] W. C. Schroeder, "Errors in Determination of Carbonate in Boiler Waters," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 5, pp. 389–393 (1933).

which may exist between a system from which steam is being removed and a system in which true equilibrium is attained. In almost all cases reasonable agreement existed between the evaporation and equilibrium studies.

CONCLUSIONS

The function of the curves in this paper is to express in as simple a manner as possible the relation between sodium hydroxide concentration and the deposition of a solid phase containing sodium sulfate from a boiler water during extreme evaporation.

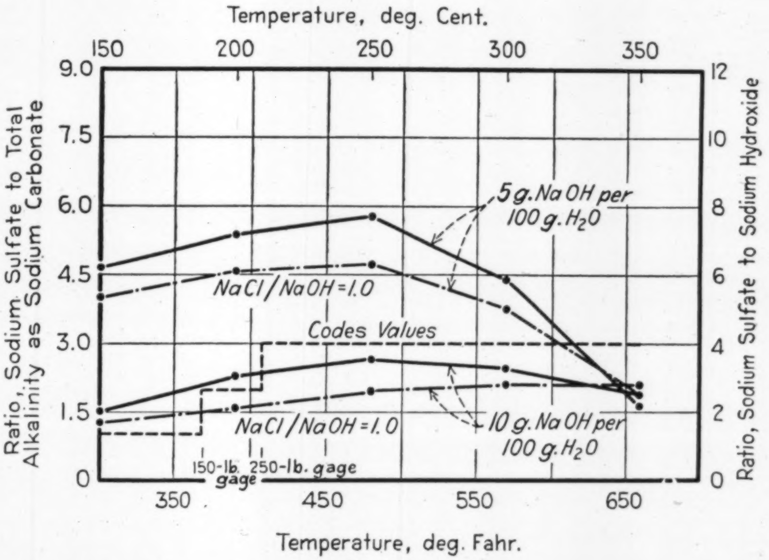

FIG. 6.—Comparison of Boiler Code Values for the Deposition of Sodium Sulfate with the Values Obtained from Complete Solubility Curves at Two Assumed Limiting Concentrations of Sodium Hydroxide.

At present, as has been noted, the curves cannot be used for the direct calculation of ratios for protection from embrittlement unless assumptions are made concerning the lowest concentration of sodium hydroxide which will attack steel. Data which are now available indicate that the lowest concentration which will produce a measurable effect is between 5 and 10 g. per 100 g. of water. These values may be subject to change with further experimental work, especially at pressures below 300 and above 1000-lb. gage. If tentatively, however, they are assumed as approximately correct, the ratios obtained from the solubility data may be compared with the ratios recommended in the Boiler Code.[7] Figure 6 shows the results of this comparison. With the assumed limiting concentration of 5 g. per

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[7] Suggested Rules for Care of Power Boilers, Section VII, A.S.M.E. Boiler Construction Code, Combined Edition, p. 442 (1935).

100 g. of water, the code values appear to be too low except for operating temperatures above 620 F. (325 C. approximately 2000-lb. gage). This is true even for solutions containing a concentration of sodium chloride equal to the sodium hydroxide. With an assumed limiting concentration of 10 g. of sodium hydroxide per 100 g. of water, the code values still appear to be low at 150-lb. gage, good agreement exists at 250-lb. gage, and the code values are slightly high above 250-lb. gage. The effect of chloride at this concentration also reduces the amount of sulfate necessary to cause deposition at all except the highest temperatures.

The work now being carried out is directed toward the determination of the limiting values for the sodium hydroxide concentrations, which Fig. 6 shows to be so seriously needed. When the values are obtained, however, it must be understood that the maintenance of the correct ratio value cannot guarantee complete protection from cracking of boiler steel. A great deal of the effectiveness of the protection will depend on the manner in which sodium sulfate or other deposited solid produces protection and the manner in which the cracking or failure happens to be produced. Both of these questions are being subjected to intensive experimental investigation at the present time.

DISCUSSION

Mr. R. E. Summers[1] (*presented in written form*).—The authors are doing much to develop understanding of the evasive type of boiler-metal failure generally indicated by the term embrittlement. Even when the practical mechanism of embrittlement is discovered a full understanding thereof, and sure methods for control of the phenomenon, must depend on just such fundamental data. The paper is especially to be commended in that it considers the possible concentrated embrittling solutions rather than only boiler waters from which those may be generated.

While we are bending every effort toward gaining knowledge of crystalloidal behavior of agents that cause or prevent metal embrittlement, let us not overlook possible colloidal action. The writer has often regarded introduction of colloid chemistry into boiler-water problems as a poor means for the disposal of unanswered questions. With discovery of the apparent role of siliceous matter in embrittlement the possibility of colloidal reactions cannot be disregarded.

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[1] Assistant Professor of Mechanical Engineering, Oregon State College, Corvallis, Ore.

THE TESTING OF ORGANIC FINISHES

By A. E. Schuh[1]

Synopsis

A method of testing organic finishes is described wherein their physical characteristics, such as distensibility, impact resistance, and abrasion resistance are quantitatively measured. When these tests are periodically performed under conditions of controlled temperature and humidity with finish specimens of controlled thickness, a means is provided for following the progressive changes with time of any organic finish. These changes measure the durability of a finish and precede the visual effects revealed by the customary method of exposure testing. A method of accelerated testing is thus provided which does not so much depend on modifying the severity of the degradative exposure factors in order to hasten ability to select better finishes, but depends on its rapidity of evaluation by providing sensitive tools for revealing signs of weakening in the earlier life of the finishes. Instead of employing detached films of finish for the physical tests, actual finish specimens, that is, finish plus base material, are used so as not to neglect the important effect of the adherence factors on finish performance. The operation of the several tests is described and demonstrated. The significance of the tests in terms of finish serviceability is discussed.

The customary methods of evaluating organic finishes by exposure testing are definitely inadequate for the problem of selecting finishes designed for long service life indoors. In an attempt to overcome this inadequacy, there has been developed by us a method of testing organic finishes based on following quantitatively the progressive effect of age by periodic measurement of the physical characteristics which define the serviceability of a finish. Thus finish specimens are subjected to scrutiny from their very infancy and a complete record of their life history is obtained.

Some years ago H. A. Nelson[2,3] and more recently Hunt and Lansing[4] have shown the value of measuring changes in distensibility of finishes with age as an index of durability. Instead of using detached films, we prefer to use actual finish specimens, that is, base material plus finish, for the measurement of such physical properties as abrasion and impact resistance and distensibility. The significance of these properties and devices for their measurement will be described.

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[1] Physical Chemist, Bell Telephone Laboratories, Inc., New York City.

[2] H. A. Nelson, "Stress-Strain Measurements on Films of Drying Oils, Paints and Varnishes," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 1111 (1921).

[3] H. A. Nelson and G. W. Rundle, "Further Studies of the Physical Properties of Drying-Oil, Paint and Varnish Films," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 356 (1923).

[4] J. K. Hunt and W. D. Lansing, "Coating Composition Films," *Industrial and Engineering Chemistry*, Vol. 27, p. 26 (1935).

In order to make the study of physical changes on finish specimens possible, it became necessary to control two very generally operating variables. These are thickness of coating and temperature and humidity of the specimens at the time the tests are performed.

CONTROL OF TEST PANELS

The importance of thickness control of test panels is illustrated in Table I. Practically every type of finishing material exhibits a pronounced thickness coefficient with respect to the various physical characteristics tested. At first glance, the magnitude of this thickness effect may seem surprising. It must be borne in mind, however, that the materials used for organic finishing owe their usefulness to the ability of their chemical components to form films by virtue of the interlocking of macromolecular groupings that are either formed in the process of oxidation or originally present. The wet-finish coatings on drying and aging undergo a progressive internal structural change. The density of the coating increases and shrinkage stresses are set up. If now, during this progressive solidification process, one whole side of the film is not free to move by virtue of the adhesional forces anchoring it at every point to the substrate surface, a

TABLE I.—RELATION BETWEEN THICKNESS AND PHYSICAL CHARACTERISTICS OF COATINGS.

Material	Abrasion Value, g. per 0.001 in.			Impact Value, r.p.m.			Pfund Hardness			Distensibility, percentage elongation		
	I[a]	II[b]	III[c]	I[a]	II[b]	III[c]	I[a]	II[b]	III[c]	I[a]	II[b]	III[c]
25-gal. ester-gum varnish												
0.0005 in.	28	17	26	>1500	900	>1500	425	640	410	>60	25.5	>60
0.0035 in.	45	41	40	900	255	1055	<5	7	6	>60	>60	>60
Air-drying synthetic A												
0.0005 in.	25	25	30	<185	<185	320	322	600	435	>60	13.4	36.6
0.001 in.	32	42	44	300	<185	555	68	500	270	>60	50.3	56.3
Air-drying synthetic B												
0.0005 in.	50	24	55	>1500	<185	1500	612	710	690	58.4	30	42.4
0.0035 in.	58	59	60	670	400	1435	110	220	140	55.5	14.8	58.3
Lacquer A												
0.0005 in.	24	21	23	>1500	>1500	>1500	875	760	880	25.5	3.0	14.5
0.003 in.	24	23	24	>1500	1500	>1500	370	480	425	36.1	18.2	19.0
Lacquer B												
0.0005 in.	52	31	51	>1500	>1500	>1500	850	820	860	19.4	2.9	13.7
0.0025 in.	57	52	54	>1500	900	>1500	570	620	550	9.4	1.7	2.4
Baking synthetic												
0.0005 in.	54	24	58	>1500	<185	>1500	525	960	480	58.6	3.1	>60
0.003 in.	70	64	72	>1500	400	>1500	36	175	41	>60	29	>60
Baked Japan												
0.001 in.	60	41	63	>1500	>1500	>1500	530	780	740	24.2	8.8	21.3
0.0055 in.	64	62	58	>1500	1500	>1500	265	400	350	4.2	3.3	2.6

[a] Aged 1 month indoors.
[b] Additional 13 months on roof.
[c] Aged 1 month indoors plus 15 months indoors at 77 F. and 50 per cent relative humidity.

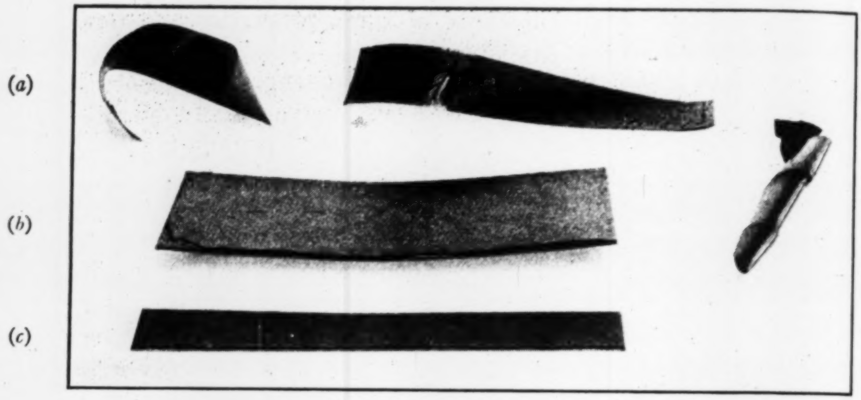

FIG. 1.—Mechanical Effect of Shrinkage Forces of Lacquer Coatings.

(a) Same lacquer as in (b) on thin copper foil.

(b) Lacquer of high tensile strength and low distensibility applied in thick coating on $\frac{1}{32}$-in. thick copper panel, showing appreciable bowing on drying.

(c) Lacquer of low tensile strength and high distensibility applied in thick coating on thin copper foil showing no effect on base material.

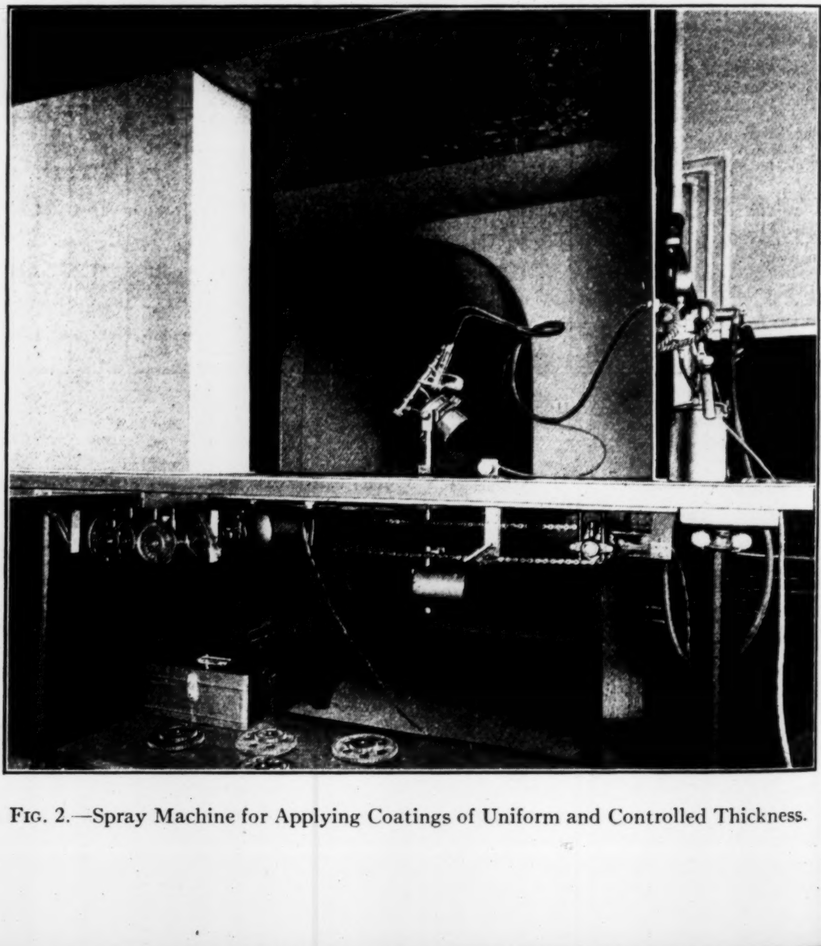

FIG. 2.—Spray Machine for Applying Coatings of Uniform and Controlled Thickness.

condition of internal strain is developed, the magnitude of which for any one material is dependent on thickness of coating.

In this connection it might be pointed out that much of the so-called adhesional failure of paint coatings may be attributable to the action of shrinkage forces. When a thick layer of gelatin is allowed to dry on a glass plate, it will be found to pull itself off spontaneously in a short time. However, a part of the glass structure is frequently pulled away with the gelatin film, showing that the force of adhesion exceeds the cohesional forces holding the glass together. Quite possibly the flaking off of very thick coats of paint can be accounted for by the same mechanism which operated in the case of the thick gelatin film. In a recent study it was observed that when a thick coating of a nitrocellulose lacquer, which in the free film evidenced high tensile strength and low distensibility, was applied on a metal panel that was $\frac{1}{32}$ in. thick, it actually bowed up this panel on drying as shown in Fig. 1.

TABLE II.—EFFECT OF TEMPERATURE AND HUMIDITY ON DISTENSIBILITY OF FINISHES.

Material	Elongation at Indicated Relative Humidity at 77 F., per cent			Elongation with Indicated Temperature at 50 per cent Relative Humidity, per cent		
	26 per cent	50 per cent	90 per cent	55 F.	77 F.	95 F.
10-gal. ester-gum varnish	1.5	4.2	49.9	0.6	4.2 2.1[a]	54.0 12.0[a]
Synthetic air-drying enamel	15.0	15.1	54.9	2.3	15.1 2.2[a]	53.2 3.8[a]
Highly plasticized lacquer	22.5	24.5	31.2	12.9	24.5 10.8[a]	31.6 25.7[a]
Baked synthetic enamel	43.0	42.3	46.6	12.5	42.3 6.0[a]	48.9 9.9[a]

[a] Coating aged 16 months indoors. In all other cases coating aged 2 weeks. All are approximateley 0.001 in. thick.

Since thickness of film is an important variable, a method of controlling it is necessary. At this laboratory the Walker spinning-disk method has been used extensively. More recently there has been developed a spraying device[5] (shown in Fig. 2) for applying coatings of controlled thickness. A spray gun is mounted on a moving arm, the speed of which is controlled over a twenty-five - fold range. Test panels are laid horizontally, one behind the other, in the path of the spray. Coatings of any desired thickness with variations of less than 0.0003 in. are readily obtained.

The importance of testing the finish coatings under conditions of constant temperature and humidity cannot be overemphasized. Practically all physical properties show a high sensitivity to temperature and humidity change. The effect of change in temperature and humidity on distensibility is shown in Table II for several types of finishes. The effect diminishes with time, but is still evident after 16 months of aging indoors. Hunt and Lansing[4] have shown in their free-film work that the reduction in sensitivity to the effects of heat and humidity is closely related to intrinsic durability. The same relationship can be shown by measuring distensibility of actual finish specimens. Examination of Table II shows it would be futile to attempt to measure physical changes of coatings without first permitting the test specimens to equilibrate to fixed positions on the temperature and humidity scale.

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[5] H. G. Arlt, "Paint Films of Controlled Thickness," *Bell Laboratories Record*, Vol. XIV, p. 216 1936).

In conducting an investigation in which a number of different samples are to be evaluated, it is customary to prepare several sets of panels of each material. An initial set of determinations by the various tests to be described briefly is then made, usually allowing at least one week between panel preparation and initial testing. After preparation, the first set of test panels is brought into a room of constant temperature and humidity for 24 hr. and tested. The rest of the sets of panels are then exposed. The type of exposure selected depends on several factors, such as the particular use for the finish, or urgency for selection. The exposure may be an indoor environment with temperature and humidity either held constant, or permitted to vary. For certain studies, outdoor exposures or an accelerated weathering test may be used. In all cases, the entire series is exposed under the same conditions for graduated periods of time. Obviously, the speed with which the physical characteristics undergo change is a function of the exposure environment; not so obvious but nevertheless important is the fact that large and significant physical changes in coatings take place long before visual signs of failure occur. However, the changes in physical characteristics are closely related to final visual exposure failure. Thus a means of accelerated testing is provided, which does not rely so much on speeding up the rate of degradation by selected exposure conditions as it does on providing tools for detecting changes during the earlier and as yet serviceable life period of the coatings.

TEST METHODS AND THEIR SIGNIFICANCE

Several of the test devices in use at this laboratory have been described elsewhere[6,7,8] and will be here referred to briefly. The abrasion test utilizes the cutting action of finely divided carborundum dispersed in a stream of air at controlled velocity. The test specimen is mounted so that it faces

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[6] A. E. Schuh and E. W. Kern, "Measurement of Abrasion Resistance," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, p. 72 (1931).

[7] A. E. Schuh, "The Evaluation of Industrial Finishes," *Industrial and Engineering Chemistry*, Vol. 23, p. 1346 (1931).

[8] A. E. Schuh and H. C. Theuerer, "Physical Evaluation of Finishes," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 6, p. 91 (1934).

the air-carborundum stream in a duplicable position, and in a few minutes a small spot appears in which the particles of highest speed have cut their way through the coating. The weight of carborundum is determined, the film thickness near the wear spot measured, and the result expressed in terms of grams of carborundum per unit thickness of film. A film thickness typical for the finish under test is used. In the average determination, several million particles of abrasive perform their momentary function and the air stream serves to maintain constant temperature in the abrading zone. This point is important owing to the high temperature coefficient of abrasion for practically all organic finishes. Since any one particle carries away only a minute quantity of the film, the factor of adherence of the coatings plays an almost negligible part. The test is highly quantitative, being limited in its precision primarily by the ability to control and measure thickness of coating.

Regarding the significance of the test, it appears to be a measure of the work required to displace completely one part of a film from another, that is, to overcome effective cohesion. Perhaps another way of stating the same thing is that it appears to be a measure of the area under a stress-strain curve where tensile strength is plotted against elongation up to point of rupture. Thus a film of high tensile strength but low elongation may show the same abrasion value as a material of low tensile strength but high distensibility. Hence the wear index, in conjunction with measurements on distensibility, appears to offer a measure of the effective tensile strength of the coatings. A detailed study in which the stress-strain relations of free films are compared with the abrasion and distensibility values on finished test panels would be worth while. It has been observed that coatings combining low abrasion value with low distensibility fail on exposure by checking and cracking. The combination of these two tests, particularly when the rate of change with time is considered, serves as an excellent means of judging durability as a function of the fundamentally important "intrinsic" film characteristics.

Distensibility is measured by bending test panels over a series of mandrels ranging stepwise in diameter from $\frac{1}{8}$, $\frac{1}{4}$, $\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$, and 1 in. With panel thickness and mandrel radius known, a value of percentage elongation can be calculated. Recently, the method has been compared with a second method of distension in which the coatings are applied on regular A.S.T.M. tension test specimens[9] for sheet metals and stretched until first signs of rupture of the organic coatings appear. The latter method has the advantage of permitting any degree of distension up to the point of rupture of the supporting base to be measured. It is particularly in the range of low distensibilities that the mandrel test does not at present offer sufficient opportunity for discrimination. Just how much of a part adhesion plays in the effective distensibility of organic finishes is still a research problem. Test specimens, after having been subjected to the mandrel test, vary greatly in their tenacity of adherence.

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[9] Standard Methods of Tension Testing of Metallic Materials (E 8-33), 1933 Book of A.S.T.M. Standards, Part 1, p. 949.

The concept of "adhesion" of paint coatings is greatly in need of clarification. Its importance is universally accepted, but what the paint technologist refers to as "adhesion" encompasses in fact a variety of undefined "adherence" phenomena. To the physical chemist, adhesion implies a force acting over molecular distances at the paint-substrate interface. This, however, is only one of several factors determining adherence of paint coatings. Until the magnitude and significance of the factors that contribute to the net adherence of paint coatings are more clearly understood, the paint technologist will have to remain satisfied with an empirical type of test which measures the ease of removal of the coating from the base material, or more precisely, the ease with which the base material is visually revealed under the action of some controlled force.

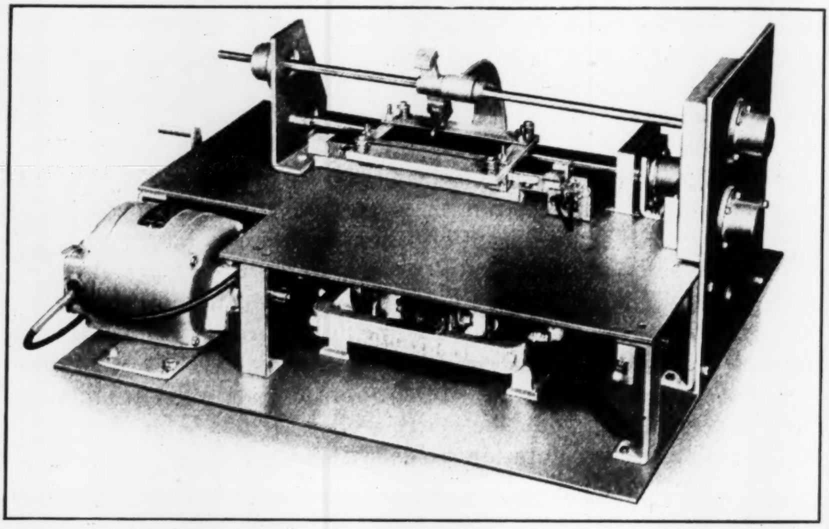

FIG. 3.—Impactometer.

One device for accomplishing this is the impact test developed at this laboratory. In this test, a freely pivoted hammer strikes a glancing blow at controlled speed on a rigidly clamped test specimen. The device, shown in Fig. 3, can be operated so as to determine either the resistance to deformation by repeated impacts at the same point, or else the maximum impact the finish can stand under increasing energy of impact. As a rule, the latter is of more interest. With the plastic type of finish, the base material is revealed by a gradual squeezing away of the paint film, whereas in the brittle or strained type of finish, a considerable area beyond the point of impact is shattered off.

With a given deformability of coating, the impact value appears to be a fairly direct measure of the adherence of the coating. The point is illustrated in Table III, where the effect of varying the nature of the base surface is reflected in the impact values.

Frequently, in the course of exposure, the bond to the base material undergoes a change owing to interfacial chemical reactions with the base material. In the case of a medium and a long oil varnish applied on brass and exposed outdoors, the impact values suddenly fell off from a value of about 1000 r.p.m. of the impacting hammer to about 300 r.p.m., even though the coating did not show embrittlement at the time, nor did similar panels exposed indoors for a prolonged period exhibit this effect. This indicated chemical attack of the base metal on outdoor exposure with attendant loss in adhesion, which was confirmed by identification of metallic compounds on the under side of the varnish films.

TABLE III.—EFFECT OF SURFACE PREPARATION ON ADHERENCE OF FINISH AS SHOWN BY IMPACT RESISTANCE.

All coatings approximately 0.001 in. thick. For finishes G and H, rotational speed of hammer was 600 r.p.m.; in all other finishes, 400 r.p.m.

		Steel		Aluminum		Phenol Fiber		Zinc			
		Smooth	Sand-blasted	Smooth	Sand-blasted	Smooth	Sand-blasted	Sand-blasted	Chemical Treatments		
									Method A	Method B	Method C
Brittle materials 3 weeks old	A	30	460	40	210	60	100				
	B	6	22	6	31	53	55				
	C	40	90	100	210	90	270				
Plastic materials 3 weeks old	D	280	310	700	1190	570	240				
	E	40	500	70	230	200	390				
	F	830	3000+	1200	3000+	3000	3000+				
2 weeks old	G	—	—	—	—	—	—	133	88	149	100
15 months old	G	—	—	—	—	—	—	44	27	12	15
2 weeks old	H	—	—	—	—	—	—	129	250+	250+	250+
15 months old	H	—	—	—	—	—	—	22	32	39	65

DISCUSSION OF TEST PROGRAM

Before discussing the general merits of the testing program, it may be worth while to mention some of the practical aspects. A single 3 by 9-in. panel is sufficient to permit the determination of the various physical characteristics mentioned. Owing to the care exercized in panel preparation, a panel of such size is sufficient to characterize reliably the finishing material under examination. One investigator can conveniently determine thickness, abrasion value, impact resistance, and distensibility of about ten different materials in one working day. In the numerous studies conducted at this laboratory within the past five years, it has invariably been found that various finishing materials submitted for any one purpose exhibit a wide spread in the values obtained by the different tests. In practically all cases, it is feasible to eliminate an appreciable fraction of the materials under test after the first test period. The feasibility of this preliminary elimination is based on the well-founded observation that organic finishing materials do not improve with age. As a rule, the test series after three months of exposure will have revealed sufficiently wide changes in slope from the initial values, that a further large fraction can safely be weeded out, frequently leaving such a limited number of suitable materials as to obviate the need for further testing.

It may seem too optimistic to expect that a testing method involving exposures of only a matter of months can be used to foretell the durability of finishes in terms of years, but it must be kept in mind that the primary road to failure for indoor finishes seems to be by way of changes within the coating itself rather than at its surfaces. There are, of course, cases where an indoor finish besides meeting the physical requirements for durability, must in addition be able to resist specific requirements, such as acid resistance, electrical insulation, or perspiration resistance. In such cases, tests for the specific requirements are performed in conjunction with the physical tests.

In the case of finishes designed for outdoor exposure, several additional environmental factors may operate to alter the course and rate of degradation of a finish. Thus sunlight may bring about undesirable color and surface changes of a finish, and rain may expedite the rusting over steel or affect the adhesion over zinc. Nevertheless, a finish to be durable must show good retention of those desirable physical characteristics measured by the tests that have been described. By performing these tests periodically on panels exposed outdoors, a much more complete understanding of the processes of finish degradation can be obtained than can be had by the present custom of observing visual changes only.

SUMMARY

A method of testing organic finishes has been described which follows quantitatively changes in the ability of coatings to conform to various controlled and significant deforming stresses. By performing the various tests with actual coatings over deformable base materials, the important effect of adhesion of the coating to the substrate is not neglected. The method is suitable, both in theory and practice, for the evaluation of organic finishes to be used indoors. For exterior finishes, this method when coupled with outdoor exposure tests, serves to clarify our understanding

of the mechanisms which operate in finish degradation. It is not necessary to await visual evidence of failure to evaluate the quality of a finish. The widest field for application of this method of evaluation will probably be found by the manufacturer or formulator of finishing materials, inasmuch as small changes in formulation may be quickly related to coating performance.

Acknowledgment.—The author gratefully acknowledges the help of his several collaborators.

DISCUSSION

Mr. H. A. Nelson.[1]—To me this is a very interesting paper because it covers a type of information that is very much needed at this time. I note Mr. Schuh's comment that in the hands of a man who knows organic finishes, such tests are very useful. I hope we can look forward to the day when we can put methods of this type in the hands of the average consumer who wants to set up service specifications. I think, however, that Mr. Schuh is wise in limiting their scope for the present.

It is worth while to find there is a correlation between progressive changes in distensibility and impact resistance and service qualities, but it should be emphasized that Mr. Schuh's results refer only to service on metal. Our own attempts to correlate such individual physical tests directly with service on wood, as in the case of house paint, have not been very successful. Apparently, too many factors enter into the picture simultaneously in the case of service on wood, such as (1) surface shrinkage and (2) total shrinkage, which I think has an entirely different effect from the surface shrinkage; (3) the cohesional strength of the film, which is indicated by the length of the stress-strain curve; and (4) specific adhesion qualities on the particular wood in question. Then add to this changes in distensibility of the film and the varying dimensional changes in the wood and it all makes a nice "pot pourri" that greatly complicates the problem of determining the exact significance of any single physical test on the paint film, such as rate of change in distensibility.

I should like to call attention to one very fundamental difference between what are known as accelerated weathering tests and physical tests of the kind discussed by Mr. Schuh. In the latter case, we are taking the results of a number of individual physical tests and attempting to interpret by summarizing or integrating the results according to the operator's experience and thus indirectly get an estimate of the quality of the paint. On the other hand, the idea of an accelerated weather test is to have the weathering unit with its combined action of destructive agencies do the integration so that the results are finally more or less obvious as in outdoor weathering. It remains to be seen which scheme proves to be the most desirable in the end. We certainly cannot take a definite position one way or the other at present, except to suggest the desirability of combining the two ideas.

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[1] Chief, Paint and Ceramic Research Division, The New Jersey Zinc Co., Palmerton, Pa.

I think Mr. Schuh has done well to point out the importance of differences in susceptibility to changes of humidity and temperature. The humidity and temperature coefficients of films are of great importance, and I think that their importance has been too generally ignored in developing new finishes.

Referring to the effects of differences in thickness on distensibility, it should be kept in mind that the effects observed are evidence of qualities common to all engineering materials when they are stretched. The difference is usually minimized if the speed of stretching is so regulated that the rate is the same per unit of cross-sectional area for both the thick and thin films. If this adjustment is made, it is usually found that differences in the paths of the stress-strain curves of thick and thin films are considerably minimized.

MR. W. R. FULLER[2] (*presented in written form*).—Mr. Schuh has given but little discussion of the data incorporated in Table I of his paper, doubtless because of space limitations. It seems probable that some of the value of these data will be lost through casual reading and I therefore offer the following summary of some of the important points that appear to be brought out by the table:

Abrasion Value.—Abrasion values of thick films are generally greater than the values for thin films. Considering that the values are based on unit film thickness, it is obvious that thick films offer increased abrasion resistance at least proportional to the increased thickness of coating. Indoor aging of films beyond one month shows little influence on abrasion values, but exposure to exterior weathering tends to reduce abrasion values. In the writer's mind the most important data relative to abrasion are not values per unit thickness but values for the actual thicknesses typical of the materials in question. It seems that the use of unit thickness data exclusively in comparing various materials may prove misleading and that the actual data for films of typical thicknesses should first be given, followed by values per unit thickness if desired.

Impact Value.—There appears to be no clear-cut relationship between film thickness and impact value when the coatings are exposed either to interior conditions or to exterior weathering. Impact values tend to increase with interior aging beyond one month and to decrease with exterior exposure.

Pfund Hardness.—After interior aging for one month, thin films are much harder than thick films. Further aging causes little change in hardness of thin films but causes thick films to become slightly harder. Exposure to exterior weathering causes definite increase in hardness, especially marked in thick films.

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[2] Technical Director, Pratt & Lambert, Inc., Buffalo, N. Y.

Distensibility.—On interior aging, thin films and thick films have about the same distensibility. The data afford no basis for a conclusion as to the relationship between film thickness and the influence of weather exposure on distensibility. Distensibility generally decreases slightly with interior aging for longer than one month and decreases to a greater extent when coatings are exposed to exterior weathering.

MR. A. E. SCHUH[3] (*author's closure by letter*).—In reference to Mr. Nelson's comments, we have recently found, as might be expected, that distensibility of organic coatings varies with rate of distension. This is particularly apparent with coatings of high distensibility, which in general means coatings that have aged but little. The effect is much more evident when the distensibility test is conducted by direct extension of painted specimens rather than by bending them over mandrels.

In commenting on Mr. Fuller's remarks, we have recently completed a study in which the factor of thickness on the physical and exposure behavior of organic finishes is critically undertaken. Thickness has been found to be an important variable and the results of this study will be published in the near future.

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[3] Physical Chemist, Bell Telephone Laboratories, Inc., New York City.

METHODS OF DETERMINING GLOSS[1]

By Richard S. Hunter[2]

Synopsis

Glossiness is evidenced by almost every object one sees and is to be attributed to specular reflection. Specular reflection occurs at the surfaces of reflecting objects and, because of the diversities of minute surface structure, many kinds of glossy appearance result. An attempt to classify these glossy appearances has led to the description of six different kinds of gloss which are named and identified as follows: (1) specular gloss, identified by shininess; (2) sheen, identified by surface shininess at grazing angles; (3) contrast gloss, identified by contrasts between specularly reflecting areas of surfaces and other areas; (4) absence-of-bloom gloss, identified by the absence of reflection haze or smear adjacent to reflected high lights; (5) distinctness-of-reflected-image gloss, identified by the distinctness of images reflected in surfaces; and (6) absence-of-surface-texture gloss, identified by the lack of surface texture and surface blemishes.

In the past, devices for measuring gloss have been developed by simple empirical means. By trial and error, methods have been found to measure the gloss of particular types of materials exhibiting particular types of gloss. It is suggested that the designer of a prospective glossmeter should determine from goniophotometric data taken on representative samples what differences in apparent reflectance are most characteristic of the differences in glossiness observed visually. That is, glossmeter design will have considerably less of the trial-and-error element when goniophotometric data are used to indicate the most pertinent reflectance measurements to make for various purposes.

Descriptions of typical gloss instruments are included in this paper, together with descriptions of the measurements they make, and a bibliography on gloss. Differences between the various types of gloss are analyzed in some detail.

Introduction

Gloss is possessed by most materials encountered in everyday life and its occurrence is so common that persons seldom stop to give it particular notice. People are, nevertheless, responsive to the general appearance of objects and nearly everyone is able to form opinion as to the beauty, attractiveness, or striking appearance of things they see. Gloss being one of the major factors in appearance, a number of methods for determining it have been developed. The purpose of these methods is usually to enable the producers and vendors of articles in commerce to rate them according to glossiness.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Physical Science Aid, National Bureau of Standards, Washington, D. C.

Definitions:

It should be noted that the term "gloss" is used in this paper in a general sense to include all characteristics such as "shininess," "sheen," "luster," etc. Following Jones (**24**),[3] *gloss* and *glossiness* are here defined so that the actual physical properties of surfaces responsible for their glossy appearance may be distinguished from the appearance itself. That is, the *gloss* of a surface is considered to be a property of the surface; *glossiness*, the appearance that results because the surface possesses that property.

1. The *gloss* of a surface is its power to reflect light specularly. Since, unfortunately, specular reflectance is a quality which may not be separated by any objective measurement from diffuse reflectance in any but special cases, this definition of gloss does not, in general, describe a quantity that can be unambiguously measured until, in addition, the conditions of measurement are precisely stated. (See section on What Glossmeters Measure.[4])

2. The *glossiness* of a surface is the appearance which results from its power to reflect light specularly. For any given surface, glossiness may vary with conditions of illumination and directions of view, but gloss is considered to be an inherent quality.

3. *Specular reflection* is that kind of reflection which causes surfaces to exhibit high lights and to appear somewhat like a mirror. This definition of specular reflection describes the process in terms of the appearance it produces and consequently does not explain the physical cause of gloss. Specular reflection is commonly contrasted with diffuse reflection which is likewise defined in terms of the appearance it produces.

4. *Diffuse reflection* is that kind of reflection which causes a surface to possess lightness or darkness of some degree which may be represented on the scale of grays running from white to black. Specular reflection and diffuse reflection are constantly used in technical descriptions of the appearance of objects. Unfortunately there is no general way in which effects of these two processes may be accurately separated. It is impossible to measure specular reflectance and diffuse reflectance as separate entities in any but an approximate way. Consequently, the separate effects of the two processes cannot be rigorously specified and the two processes are, for this reason, defined in the present paper in terms of the appearance each produces, even though it is customary to think of them as physically separate processes dependent upon the structure and composition of the object. Specular and diffuse reflection are discussed further in a separate section on Specular and Diffuse Reflection.[5]

5. *Apparent luminous reflectance*, hereinafter termed *apparent reflectance*, is defined as the luminous reflectance a perfectly diffusing surface

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 803.

[4] See p. 792.

[5] See p. 791.

must have in order to yield the same brightness as the unknown surface under the same conditions of illumination and viewing (35). That is, any surface observed in any manner and illuminated by any combination of illuminants is compared to the theoretical, perfectly reflecting, perfectly diffusing surface observed and illuminated under exactly the same conditions. (See Fig. 2 and section on Directional Distribution of Reflected Light.[6]) In practice, of course, the theoretical, perfectly reflecting, perfectly diffusing surface is not obtainable, but material standards of known apparent reflectances may be obtained in several ways. For many cases, relative apparent reflectances are all that are needed and standards of known apparent reflectance are not required.

TABLE I.—CLASSIFICATION OF THE SIX TYPES OF GLOSS.

Type of Gloss	Appearance Characteristics Produced	Most Often Used to Describe Appearance of	Gloss Range	References in Bibliography
Specular gloss (formerly objective gloss)	Brilliance of specularly reflected light, shininess	Paints, surfaces of moderate gloss, dark and chromatic objects	Medium gloss	**3, 5, 14, 16, 17, 23, 31, 32, 39, 43, 44, 47, 52, 54**
Sheen	Shininess at grazing angles	Flat paints, papers, and materials of low gloss	Low gloss	**37, 44**
Contrast gloss (formerly subjective gloss)	Contrast between specularly reflecting areas and other areas	Papers, matte and semimatte finishes, white and light-colored materials	Low gloss	**7, 8, 9, 11, 12, 15, 22, 24, 25. 26, 27, 28, 29, 33, 34, 44, 45, 46, 48, 49**
Absence-of-bloom gloss	Absence of smear or excess semi specular reflection adjacent to reflected high lights and images	Surfaces in which reflected images and high lights may be seen	High gloss	**21, 26**
Distinctness-of-reflected-image gloss	The distinctness and sharpness of reflected images	Finishes, enamels, lacquers, and all smooth image-reflecting surfaces	High gloss	**10, 18, 21, 43, 51, 53**
Absence-of-surface texture gloss	Surface evenness, absence of texture, indicated by difficulty of recognizing presence of surface	Glossy materials, finishes, and coatings	Medium to high gloss	**17**

The plan of the present paper is to describe first the six different types of gloss in terms of the appearances they produce. Following this is a discussion of the directional distribution of light reflected by surfaces and the various measures thereof. With information thus at hand on the appearance characteristics resulting from the different types of gloss and on the basic method of reflectometry which is inseparable from the discussion of gloss, it becomes possible to develop relationships between these two phases of the subject.

SIX TYPES OF GLOSS

The necessity for a classification of gloss types arises when one attempts to grade materials for gloss, or to describe their differences in appearance.

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[6] See p. 787.

With two materials of the same general appearance, it is possible to say that one or the other has the higher gloss, or that the two appear about the same. For two materials of different appearance, however, it often cannot be stated which has the higher gloss. They may not show a common type of glossiness by which they may be graded.

From a study of the appearance by which the gloss of surfaces is commonly graded and from a study of the different existing gloss-measuring instruments and the properties they measure, a classification of gloss into six types has been devised. A classification was first made by the author in the fall of 1935 when, however, only five types were identified (18). Table I, which gives the classification, is a modification of the previous table and lists: (*a*) the six types of gloss, (*b*) the appearance characteristics of each, (*c*) materials with which each type of gloss is most often identified, (*d*) the position in the general gloss range in which surfaces possessing each of the types of gloss are most often found, and (*e*) references to instruments which measure each type of gloss. Figure 1, reproduced from this article (18), serves to illustrate some of these types and shows photographs of gloss-comparator images reflected in four pairs of surfaces. The first two surfaces differ essentially in specular gloss, the second two in contrast gloss, the third two in bloom, and the fourth two in surface texture. Variations in distinctness-of-reflected-image gloss are also seen; for instance, the first two samples reflect images quite accurately but the second two do not. Sheen, which was only recently added to the list, is not illustrated. Further details regarding these types of gloss, their significance and their measure-

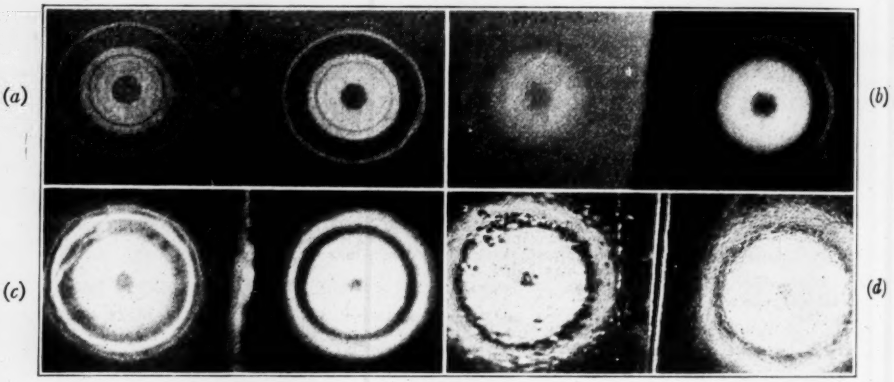

FIG. 1.—Images Photographed with Gloss Comparator (18) Showing Different Types of Gloss.

(*a*) The right image is brighter than the left image and depicts higher specular gloss.
(*b*) The right image is from a black surface, the left image from a white surface. The black surface shows higher contrast gloss.
(*c*) Although the left image is sharper, it shows reflection smear, or bloom.
(*d*) Camera focused on surfaces to record texture. Left—pimpled, right—orange peel.

ment, are given in the later sections of this paper and in other papers (18, 19, 21).

This scheme of identifying gloss types is based upon observation and upon methods of classification and grading gloss that are already in existence. It is not intended to be the final and complete method for describing gloss and glossiness. There seem to be several types of glossiness that are not adequately explained by this analysis; for instance, some of the appearances that are associated with degree of polish are particularly hard to describe in the terms given in Table I. Much more data are needed on the correlation of differences in glossiness identified by observers and the results of instrumental measures of gloss. Viewed under different conditions, surfaces can present many more than six different types of glossiness, although in many cases the more complex effects may perhaps be treated as combinations of the simpler effects described above.

The need for simplicity in the treatment of the subject must be compromised with the need for an analysis of the problem complete enough to include within its scope methods of answering most of the questions that will arise. As an indication of the adequacy of Table I, it may be noted that it served to place, according to type of gloss measured, every one of the thirty-seven methods of measuring and analyzing gloss covered in the bibliography. On the other hand, some of the effects described in the papers dealing with the phenomenological and psychological phases of the subject were difficult to represent in terms of the six-gloss-type classification.

Smooth metal surfaces exhibiting metallic glossiness are distinguished by the fact that a major portion of the incident light is reflected specularly, whereas but 3 to 8 per cent of the incident light is reflected specularly from non-metallic surfaces at the non-grazing angles. Thus a major portion of the light incident upon a non-metallic surface may be reflected diffusely, whereas metallic surfaces are commonly characterized by a relative lack of diffuse reflectance. In spite of these differences, it is believed that, inasmuch as both metallic and non-metallic surfaces exhibit specular reflectance, the above classification designed to describe non-metallic surfaces may also be used to describe metallic surfaces. At present, however, no data are at hand to show the applicability of the six-type classification to descriptions of metallic appearance.

DIRECTIONAL DISTRIBUTION OF REFLECTED LIGHT

Data describing the intensity distribution of incident and reflected light as a function of angle form the physical bases for descriptions of their gloss. Such data are commonly presented as curves or numerical values giving, for specified angular distributions of illumination, values of apparent reflectance for different directions of reflection.

In Fig. 2, two experimentally obtained reflection-distribution curves are given together with a theoretical curve for comparison. In this diagram, light from a single direction is represented by *I* and is incident upon the reflecting surface *O* at 45 deg. *S* indicates the direction of reflection of this light for the case in which the reflecting surface is optically smooth and mirror-like. The curve *D* gives the apparent reflectance for the theoretical, perfectly reflecting, perfectly diffusing surface which possesses, by definition, an apparent reflectance of unity in all directions. That is, apparent reflectance is defined as the reflectance a perfectly diffusing surface would have to have in order to yield the same brightness as the unknown surface under the given conditions of illumination and viewing. Compared to the perfectly diffusing surface, surfaces commonly reflect a disproportionately large amount of light in the general direction of mirror reflection because of gloss; consequently their apparent reflectance may rise to well above unity in that direction.

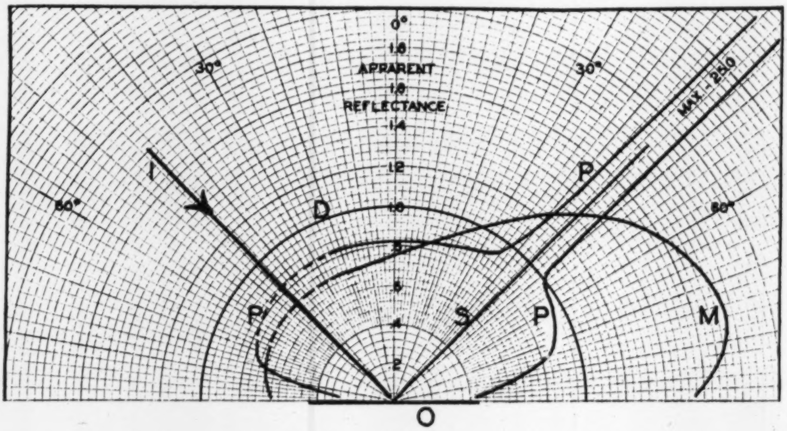

FIG. 2.—Reflection-Distribution Curves for Porcelain-Enamelled Plate *P* and Sheet of Mimeograph Paper *M*.

Direction of incident illumination *I*, direction of mirror reflection *S*, reflecting surface *O*, and reflection distribution for ideal, completely reflecting, perfectly diffusing surface *D* are also indicated.

Curve *M* gives the apparent reflectance for a nearly-matte sample of mimeograph paper; and *P* the apparent reflectance for a vitreous, porcelain-enamelled plate. As would be expected, the vitreous porcelain concentrates the reflected light about the direction of mirror reflection rising to a measured apparent reflectance of 25.0 at 45 deg. In the case of the mimeograph paper, it can be seen that the apparent reflectance is higher opposite the incident illumination, but that there is no concentration of apparent reflectance in the direction of mirror reflection. In fact, it is interesting to note that the highest apparent reflectance appears in a direction further from the normal than mirror reflection. This type of curve is characteristic of many types of nearly-matte surfaces.

Because Preston[7] found that a freshly prepared, magnesium-oxide surface illuminated at 45 deg. and viewed normally possessed an apparent reflectance of 1.00, secondary standards of reflectance obtained by comparison with magnesium oxide are satisfactory standards of apparent reflectance for these conditions. When the apparent reflectance of a given surface is known for one direction of illumination and view together with its relative brightnesses for other directions of view, its apparent reflectances for these other directions may be derived because the brightness of the perfectly diffusing surface does not vary with direction of view.

The complete specification of a surface's ability to direct reflected light is so complex that it is virtually never determined. Light may be reflected by a surface in all directions; furthermore, with an infinite number of possible incident illuminations, each of which may result in a different distribution of light, the complete reflection-distribution specification for any surface is made up of a "quadruple infinity" of apparent reflectances. Instead of complete goniophotometric analyses, partial analyses such as shown by the curves presented in Fig. 2 are usually adopted. For example, only those values of apparent reflectance in the plane of the 45-deg. incident beam and the normal to the surface are given in Fig. 2. In using single curves such as these, it must be realized that they do not present in any case the complete physical basis of glossiness and that there may often be reflectance effects of importance to the investigator not indicated by the data given.

Jones **(24)** was the first to emphasize the importance of goniophotometric measurements in gloss work. McNicholas **(35)** has treated the theoretical side of the subject and, in addition, published much valuable experimental data.

In discussing reflection distribution above, it was assumed that exact values of apparent reflectance for exact unidirectional illuminations and directions of view could be obtained. Actually, it is impossible to obtain curves for true unidirectional illumination and true unidirectional viewing because every source and every receptor used in a reflectance-measuring instrument possesses finite size. It is doubtful whether there exists any goniophotometer possessing sufficiently small illuminating and viewing apertures to deal successfully with all gloss problems.

The eye is able to resolve images separated by one minute of visual arc. To equal the eye in distinguishing differences between surfaces of high gloss the goniophotometer should likewise be capable of revealing these effects in terms of brightness. It is believed that no goniophotometer has been produced up to the present time, which possesses this power of resolution. To build such an instrument it will be necessary to have the source and receptor of narrow aperture. For each determination, the light which reaches the reflecting surface from the source must follow a path in which all rays are parallel to within one minute of arc, and that reaching the receptor from the reflecting surface must satisfy the same conditions, so that, within this tolerance, one and only one direction of incidence and reflection is accepted for measurement. Only with such an instrument will it be possible to obtain sufficiently accurate reflection-distribution curves to differentiate all surfaces of high gloss.

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[7] J. S. Preston, "The Reflection Factor of Magnesium Oxide," *Transactions*, Optical Soc. (London), Vol. 31, p. 15 (1929–1930).

Most of the goniophotometers now in use accept, for measurement in one setting, light reflected by the test surface at angles comprising several degrees. Accordingly, each apparent-reflectance value obtained represents the average of apparent reflectances for all the directions included in the measurement. Therefore the distribution curves obtained with such instruments are less selective with respect to angle of reflection than are the ideal curves for unidirectional illumination and measurement. Figure 3 is a diagram of the modification of McNicholas' goniophotometer (36) constructed at the National Bureau of Standards, so designed as to give high angular resolving power. A single coil of a monoplane-filament lamp provides a source of narrow aperture. In front of the photocell receptor is an adjustable slit which can be made as narrow as desired, the only requirement being that sufficient light pass through it to give a current from the photocell that will register satisfactorily. This new apparatus was used to obtain the curves of Fig. 2. It gives a reflection-distribution resolving power of about one-half degree. The source and sample may be rotated about the axis of the sample position, but the receptor position is fixed.

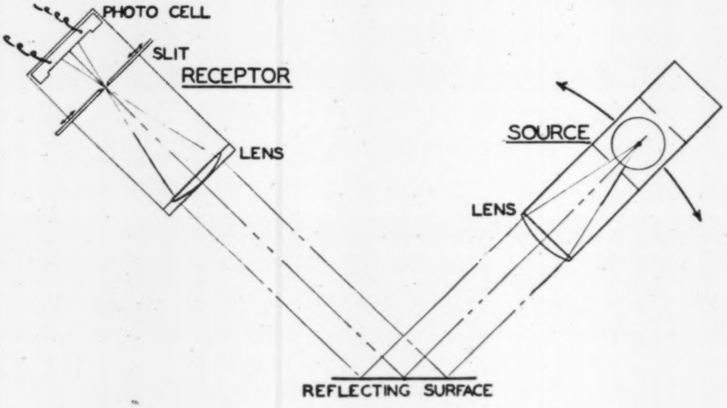

FIG. 3.—Photoelectric Modification of McNicholas' Goniophotometer with Narrow Source and Receptor Apertures (36).

The receptor is fixed in position; the source and reflecting surface may be rotated about the center of the reflecting surface, either separately or together.

SPECULAR AND DIFFUSE REFLECTION

As was pointed out in the introduction, the concepts of specular and diffuse reflection are widely used in descriptions of the appearance of opaque objects. It has, in fact, been customary to say that specular reflection is responsible for gloss, although in Table I, it may be seen that absence of diffuse reflection increases gloss of at least one type (contrast gloss).

In order to form a picture of the mechanism of reflection, one should consider the minute reflecting areas of individual particles as well as the larger visible areas of objects. Reflection occurs whenever light encounters a boundary between two media differing in refractive index.

Specular reflection is ordinarily "first-surface" reflection taking place at the initial contact of the incident light with the reflecting object—as from enamelled tile. Diffuse reflection is, in most cases, principally a combination of reflection, refraction, and Rayleigh scattering taking place within the body of the reflecting object by the pigment particles and other particles having refractive indices differing from the refractive index of the surrounding medium. Only when the surface of an object is optically smooth is it possible quantitatively to separate the specular reflectance from the diffuse, the former in this instance being confined entirely to the direction of mirror reflection. The porcelain plate, represented by curve *P*, Fig. 2, is quite smooth. From its reflection-distribution curve, it may be seen that it would be possible to divide the observed reflectance satisfactorily into two components: one, diffuse reflectance, nearly constant in all directions; and the second, specular reflectance, which is the excess in the direction of mirror reflection.

For objects whose surfaces are not optically smooth—and the majority fall in this class—it is not possible to separate the observed reflectance into its components so easily. That is, it is not possible to decide exactly what portion of the reflectance is diffuse and what portion is specular. The mimeograph paper, represented by curve *M*, Fig. 2, presents a characteristic reflection distribution in which a transition from the high reflectance in the approximate direction of mirror reflectance and lower reflectance in other directions is wholly wanting—giving no indication of a separation into diffuse and specular components. Typical of materials which possess surfaces that are not optically smooth are semi-matte painted finishes whereon protruding pigment particles or voids left after the evaporation of minute pools of volatile liquid break the smoothness of the surface; also sheets of paper in which there are voids between the individual fiber, pigment and resin particles composing the sheet. Materials such as these frequently present a glossy appearance probably ascribable to a tendency for the individual surface units to follow the surface shape of the object.

Each specimen has its own reflection distribution, and only where the surface as a whole is optically smooth is it possible to separate the diffuse reflectance from the specular reflectance. Where materials differ as much in their power to direct reflected light as the white porcelain and white mimeograph paper of Fig. 2, the question of whether one or the other is lighter is obviously a question of how they are illuminated and viewed.

WHAT GLOSSMETERS MEASURE

Most instruments designed to measure gloss actually determine some arbitrarily chosen type of apparent reflectance which has been found by experience to correlate closely with the particular type of gloss being studied. That is, most glossmeters have been designed empirically because few of their designers have had the opportunity to obtain goniophotometric data pertinent to the types of material whose gloss is to be measured.

It is advantageous for the designer of a glossmeter to determine from goniophotometric data on representative samples what particular apparent reflectance, or function of apparent reflectances, correlates best with glossiness, and then construct an instrument to measure this particular function. It is the object of research now being carried forward at the National Bureau of Standards to develop high-precision goniophotometric apparatus to study various gloss problems, to determine to what extent existing gloss-measuring apparatus is applicable to each problem, and to devise new apparatus if existing devices are found inadequate. Only Jones (**24**) is known to have made goniophotometric measurements on his materials before developing a glossmeter to measure them. The primary requirement for a glossmeter is that it give results which correlate satisfactorily with the gloss ratings on these same specimens assigned by visual inspection. In addition, the instrument should be reproducible from the description, so that the ratings obtained will not depend upon any particular instrument. Other desirable features are portability, inexpensiveness, ruggedness, and simplicity.

Certain conditions are necessary to a reproducible determination of gloss: (1) direction and aperture of both incidence and view, (2) spectral composition of the light, and (3) polarization of the light. For any method of measuring gloss, the fulfilment of the first is most important.

1. *Direction and Aperture of Both Incidence and View.*—For a complete description of a reflectance measurement, it is necessary to give the angles of incidence and reflection of all rays of light leaving the source and reaching the receptor by way of the test surface. That is, all those portions of the reflection distribution which enter the measurement must be given. Since every source of light and every receptor must possess finite size, illumination of the sample in any instrument comes, not from a single point, but from the integral parts of the source. Similarly, light leaving any one point of the surface of the sample may take any one of a number of angles and be incident upon various parts of the receptor. The elements that determine the

possible combinations of angles of incidence and reflection by which light may travel from the source to the receptor are (1) the central directions of incidence and viewing, (2) the solid angular apertures of source and receptor, and (3) the area of the surface tested. Thus to describe completely the reflectance indication of a given glossmeter, it is practically necessary to have a drawing to sflale of its complete optical system.

For an approximate angular specification of a gloss measurement, the factors of first importance are the angles which the axial rays of the incident and viewing beams make with the normal to the test surface. This information is available for practically all glossmeters. Thus with Jones' glossmeter (**24**), Fig. 8, two beams reflected at 0 deg. and −45 deg. are compared; the surface being illuminated at 45 deg. Pfund's instrument (**44**), Fig. 5, always makes the angle of view equal to the angle of incidence of the illumination. The Ingersoll Glarimeter (**22**), Fig. 9, measures the polarization of light reflected at $-57\frac{1}{2}$ deg., with illumination incident at $57\frac{1}{2}$ deg.

In addition to the axial directions of illumination and view, data on the angular aperture of the light within the beams is important for the description of a glossmeter. This factor is of major importance in any measurement involving specular reflection because, as can be seen from the reflection distribution curves above, the angular spread of specular reflectance varies markedly with type of glossy surface. To simplify the description of this factor so that all possible angles of incidence and view do not have to be stated, it is usually sufficient to give the maximum deviation, from the angle of mirror reflection, of any part of the beam accepted for measurement as specular reflection. To arrive at a figure fairly indicative of this maximum angular deviation from mirror reflection, one-half the sum of the angular apertures of source and receptor with regard to the reflecting surface may be taken for any instrument. In the case of the Ingersoll Glarimeter a cone of light 13 deg. in diameter is incident upon the glossy surface (see Fig. 9). With the solid angular aperture of the viewing element, an artificial pupil, negligible when compared to the 13 deg. of the source, the maximum angular deviation from the direction of mirror reflection of light accepted for measurement is $6\frac{1}{2}$ deg.

2. *Spectral Composition of the Light.*—Spectral specifications include descriptions of the following three factors: spectral distribution of energy from the source, spectral transmissions of any filters in the system, and spectral sensitivity of the viewing element. Almost without exception the instruments now being used to measure gloss employ an incandescent light source but do not use spectral filters[8] to alter the color of this source. Since the majority of instruments are visual, the usual viewing element is

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[8] In some few cases, instruments which measure contrast gloss make provision for the use of a selective spectral filter when highly chromatic surfaces are to be measured. It is advisable to do this in order to eliminate chromaticity differences from the photometric field.

a human eye and this possesses a spectral response similar to the adopted visibility function. However, several of the more recent glossmeters are photoelectric and some of these use cells whose spectral response differs quite markedly from that of the average human eye. Where such cells are used, and particularly where these possess appreciable infra-red or ultra-violet sensitivity, either the response of the cells should be corrected to approximate visual sensitivity, or the results should not be used as if they were equivalent to visually obtained values.

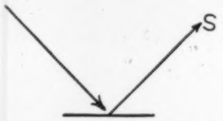

FIG. 4.—Specular Gloss Indicated by Apparent Reflectance in Direction of Mirror Reflection S.

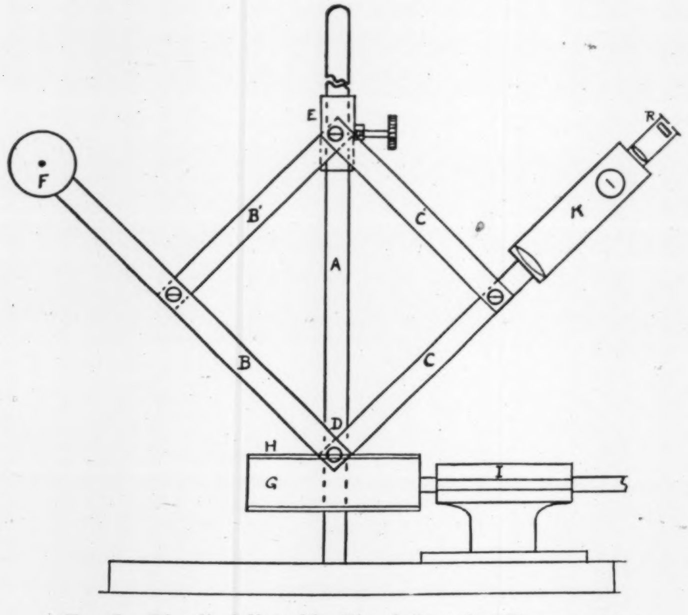

FIG. 5.—Pfund's Adjustable, Visual, Specular-Glossmeter **(44)**.

The sample is bent in the form of a cylinder over *H*. Light from the source *F* is specularly reflected by the test surface and measured by means of a pyrometer lamp *K*. It is possible to vary the angle at which measurements of specular reflection are made.

3. *Polarization of the Light.*—Because light specularly reflected at any angle other than normal is, in general, partially polarized, instruments which measure specularly reflected light must be described so that the effect of the optical components upon the state of polarization of the light will be given.

Light specularly reflected from glass at about 57 deg. is completely plane polarized. For many materials having surfaces less smooth than polished glass, the light specularly reflected at this angle is to a major degree also plane polarized. A method frequently used to measure contrast gloss (**15, 22, 25, 46**) employs a doubly-refracting prism to separate this plane-polarized specular component of reflectance together with half the diffuse reflectance from the other half of the diffuse reflectance which is plane polarized at right angles to the specular component. By means of a polarizing prism the intensities of the two separate beams may be compared and a value of contrast gloss obtained (see Ingersoll Glarimeter, Fig. 9). Instruments which employ this type of contrast measurement give results that usually differ from the contrast-gloss measurements made by other instruments.

SPECULAR GLOSS

(FORMERLY TERMED OBJECTIVE GLOSS (18, 44))

Apparent reflectance in the direction of mirror reflection is indicative of specular gloss as shown diagrammatically by Fig. 4. The shininess of a surface, the brilliance of its reflected high lights, or its specular reflectance are the appearances corresponding to specular glossiness. In this paper, the term sheen has been applied to specular gloss at near-grazing angles.

Specular gloss is probably the simplest type of gloss to determine since its measurement involves finding only the apparent reflectance in the direction of mirror reflection through an instrument aperture which is adapted to the types of surfaces studied. To determine values of specular gloss, permanent standards of apparent reflectance are used to calibrate the photometric device used to make the measurements. Pfund's Glossmeter (**44**), Fig. 5, uses black glass standards of reflectance and a pyrometer lamp for photometer. This instrument may be adjusted to measure specular gloss at any angle of reflection desired.

Specular gloss measurements are most commonly applied to materials of medium glossiness such as house paints, linoleums, printing ink, etc. On surfaces of low gloss the apparent reflectance in the direction of mirror reflection is often but little larger than the apparent reflectance in other directions (see curve *M*, Fig. 2). For such surfaces, glossiness is usually better indicated by measures of contrast gloss than by measures of specular gloss. For surfaces of high gloss where the apparent reflectance in the direction of mirror reflection is of the same order of magnitude as the apparent reflectance from perfectly-polished surfaces of the same material, one cannot distinguish differences in specular gloss as readily as differences in the distinctness of images reflected in such surfaces, or differences in bloom, or surface texture. In other words, all surfaces of high specular gloss appear shiny, but small differences in shininess do not attract attention as do the more apparent differences in other gloss qualities.

In designing any device to measure specular gloss it is desirable, on the one hand, to have the angular apertures relatively small so that the instrument receives for measurement only light that is reflected in nearly the exact direction of mirror reflection. On the other hand, surfaces that are warped or wavy furnish a difficult problem when measured by instruments of small aperture because such surfaces slightly misdirect some of the specularly reflected light so that it misses the narrow aperture and has no part in the resulting measurement. An observer grading surfaces for shininess may see reflected high lights displaced because of warpage or waviness, but he discounts these warpage effects in visual grading; consequently an instrument to make the same gradings must discount the effects also. This requires instrument apertures larger than otherwise needed. In designing a specular-glossmeter to measure house paints and other products in the range of medium glossiness, the author (16) found that a source aperture 8.5 deg. in diameter, for an angle of reflection of 45 deg., was satisfactory; the receptor aperture, an artificial pupil, was in this case relatively small.

As explained above, specular gloss measurements are measurements of apparent reflectance employing the direction of mirror reflection and as small an aperture surrounding this direction as the curvature and waviness of surfaces will permit. This makes specular gloss measurements nearly equivalent to measurements of the so-called specular reflectance so that they are less affected by the so-called diffuse reflectance than are other measurements of gloss. For this reason measures of specular gloss are used to compare the gloss characteristics of surfaces of different hues and different lightnesses. In the paint industry, for instance, it is customary to say that white, black and chromatic paints which have the same vehicular composition and thus have about the same specular reflectance characteristics, have the same gloss. A specular-gloss instrument is well suited to measure them.

SHEEN

The author was only recently introduced to sheen as a type of gloss. Undergoing examination were a number of flat wall paints, none of which appeared shiny or exhibited appreciable specular gloss at the usual angles of view. However, when these same samples were viewed at grazing angles, some appeared very shiny and others remained matte in appearance. This type of gloss was said to be of considerable importance in descriptions of the appearance of flat wall paints. Appearances of an opposite type have also been observed in the case of samples which were distinctly shiny at all angles except those near grazing. Examples have been noted of a number of yarns and paper samples which possessed a fuzziness that caused the to appear matte if viewed at near-grazing angles.

Sheen, or specular gloss at near-grazing angles, may be measured with an instrument such as the Pfund Glossimeter, which may be adjusted to near-grazing angles, or Milligan's instrument (37). The measurement of sheen is indicated diagrammatically in Fig. 6.

CONTRAST GLOSS

(FORMERLY TERMED SUBJECTIVE GLOSS (18, 44))

On surfaces of low gloss, particularly where such surfaces are white or light-colored, the high lights due to specular reflection may appear distinguishably brighter, but not greatly brighter than those areas which do not reflect high lights to the observer. When one views a surface of low glossiness, he seems to appreciate the contrasts between areas reflecting to him specularly and adjacent areas reflecting diffusely. Such a contrast measurement is indicated diagrammatically in Fig. 7. Contrast-gloss measurements are employed for materials such as paper, flat wall paint, flat lacquer, yarn, etc.

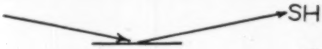

FIG. 6.—Sheen Indicated by Apparent Reflectance in Direction of Mirror Reflection at a Grazing Angle SH.

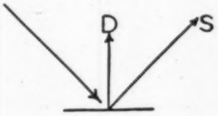

FIG. 7.—Contrast Gloss Indicated by Contrast Between Apparent Reflectance in Direction of Mirror Reflection and Apparent Reflectance in Another Direction.

Possible functions are $1 - \frac{D}{S}$ and $\frac{S}{D}$.

Since the gloss of these materials is low, the excess light due to specular reflectance is widely distributed in the general direction of mirror reflection in a manner characteristic of such surfaces (see curve M, Fig. 2). For this reason the apertures of instruments used to measure this type of gloss may be made large if desired. Two contrast-glossmeters are illustrated in Figs. 8 and 9. In Jones' instrument, Fig. 8, it may be seen that the apparent reflectance at 45 deg. is photometrically compared with the apparent reflectance at normal viewing.

In the Ingersoll Glarimeter, Fig. 9, the large source aperture (indicated by the heavy lines, 1 by 1-in. opening, $5\frac{1}{4}$ in. from the center of the sample) is equivalent to a circular opening approximately 13 deg. in diameter. Since this instrument is generally employed throughout the paper industry, it probably enjoys wider usage than any other glossmeter. The angle of reflection is $57\frac{1}{2}$ deg., the angle of polarization for paper. A doubly-refract-

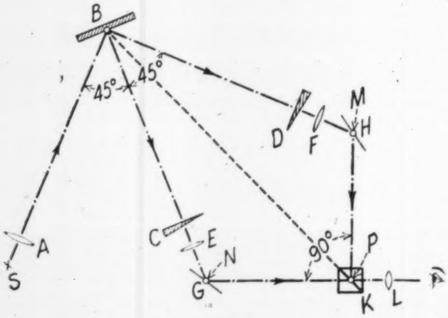
B
45°
45°
M
H
D
F
C
E
N
A
S
G
90°
P
K
L

FIG. 8.—Diagram of Jones' Contrast-Glossmeter (**24**).

Light from the source S is incident upon the sample B at 45 deg. The apparent reflectance at −45 deg. (diffuse plus specular) is brought into juxtaposition with the apparent reflectance at 0 deg. (diffuse) by means of mirrors G and H and the Lummer-Brodhun cube K. The observer can adjust the resulting beams to equal brightness by means of wedges C and D. The relative brightness of the two reflected beams is given by the inverse ratio of the transmissions of the wedges necessary to bring them to balance in the eyepiece.

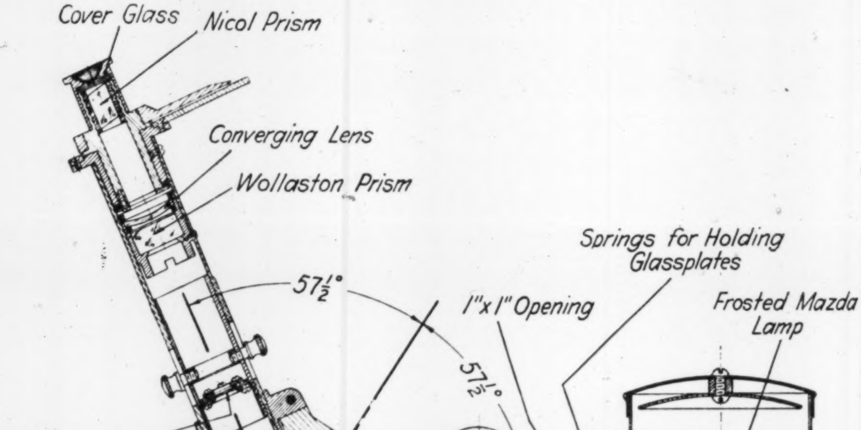
Cover Glass
Nicol Prism
Converging Lens
Wollaston Prism
Springs for Holding Glassplates
57½°
57½°
1"x1" Opening
Frosted Mazda Lamp
Adjustable Slit
Sample
5⅛"

FIG. 9.—Vertical Cross-Section Drawing of Ingersoll Glarimeter (**22**).

Contrast gloss is indicated by the state of polarization of light reflected in the direction of specular reflection for an angle of reflection of 57½ deg.

ing Wollaston prism is employed to divide the reflected beam into two beams polarized at right angles for comparison.

Absence-of-Bloom Gloss

By bloom is meant the appearance of haze or smear upon a glossy surface adjacent to a strong specularly reflected high light. Bloom appears most strikingly on dark surfaces which give by reflection relatively bright images; thus, on a highly polished automobile finish, the smear from a dirty rag or a little oil is plainly visible, particularly when the surface is viewed near an adjacent high light. But little scientific work has been done on this type of gloss although it apparently has an important influence on appearance.

This completes the list of types of gloss which are at present subject to photometric determination, and except for a little work that has been done on metallic surfaces **(26)**, little is known about the quantitative aspect of bloom, which is indicated visually by an excess of apparent reflectance adjacent to the mirror reflection. Whether, for its measurement, it will be desirable to take the difference between apparent reflectance in the direction adjacent to mirror reflection and apparent reflectance in a direction well removed from mirror reflection, or to take some other function of these apparent reflectances is not yet evident. Different possible magnitudes for indicating bloom are suggested by the diagram in Fig. 10. In any case, care must be exercised to insure that none of the strongly reflected high light is admitted as bloom.

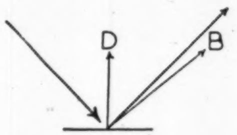

Fig. 10.—Bloom Indicated by Excess of Apparent Reflectance Adjacent to Mirror Reflection. Possible functions are $B - D$, $1 - \frac{D}{B}$, and $\frac{B}{D}$.

In Fig. 11, from another paper by the author **(21)**, is shown a comparison of the power of two surfaces to reflect images of the target used in the Gloss Lamp. The specimen on the left clearly shows bloom in the dark areas of the image.

Distinctness-of-Reflected-Image Gloss

Surfaces which give images by reflection are, in general, surfaces of high gloss. Because these images are often the most pronounced feature of the appearance of a surface of high gloss, it has been widely used as a criterion in gloss investigations. No photometric method of measuring distinctness-of-reflected-image gloss has as yet been developed, but this

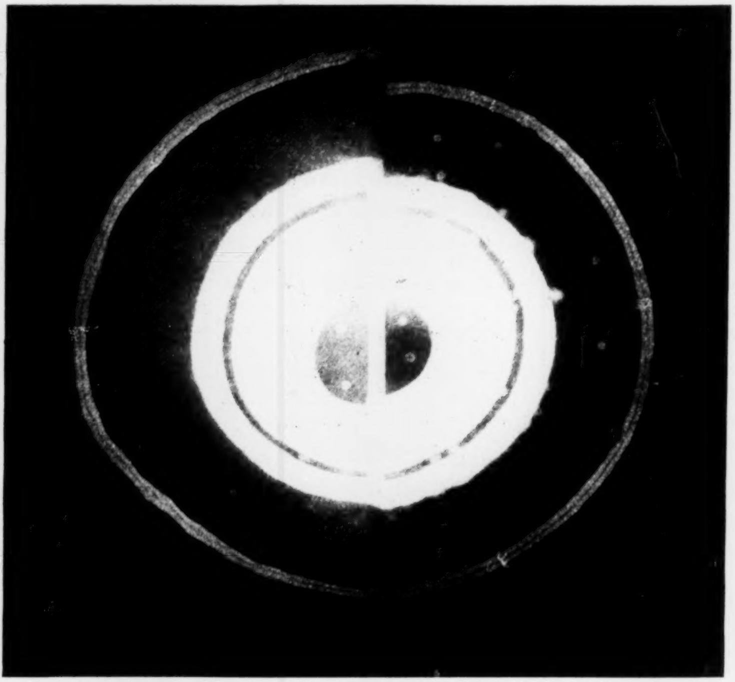

FIG. 11.—Gloss-Lamp Target (**21**) Reflected in Pair of Surfaces.

The specimen on the left shows the reflection haze due to its bloom in the dark area of the image adjacent to the bright center circle.

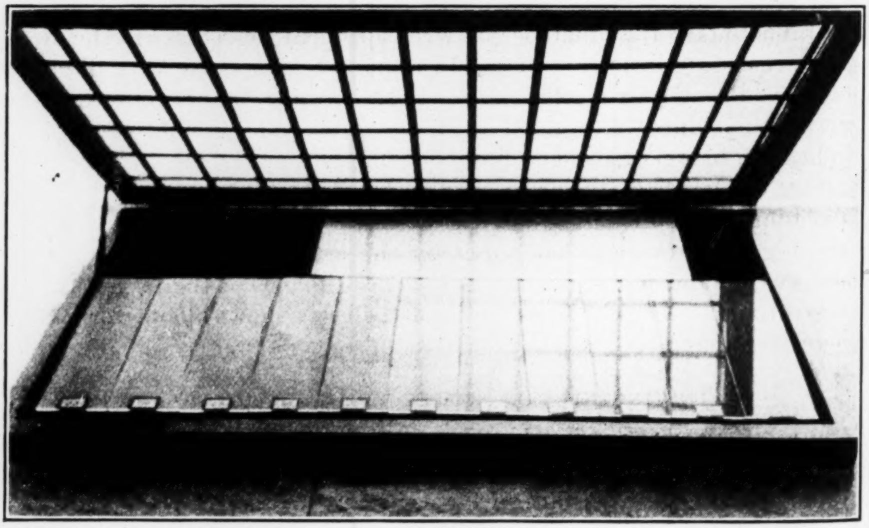

FIG. 12.—Detroit Paint Production Club Method of Rating Distinctness-of-Reflected-Image Gloss (**10**).

The series of standard lacquers of varying distinctness-of-reflected-image gloss are arranged in a row and compared to test samples.

type can probably be associated in magnitude with the steepness of the reflection distribution curve in the region of specular reflection (see curve *P*, Fig. 2). So far the methods developed to measure it depend on empirical comparisons.

Probably the method most widely used to reveal gloss differences consists in the comparison of the reflected images which two surfaces give of a window sash. This procedure is usually one of grading the specimens for distinctness-of-reflected-image gloss.

Figure 12 illustrates one attempt to establish a distinctness-of-reflected-image gloss scale on a more permanent and reproducible basis than is furnished by the window sash combined with day-to-day comparisons of samples and standard. In 1932 the Detroit Paint Production Club (10) developed a series of standard lacquers of varying degrees of distinctness-of-reflected-image gloss. These lacquers were arranged on panels under a lattice frame as illustrated in Fig. 12. The images reflected by the test sample were compared with those reflected by the members of the series of standards. Lack of permanence of the lacquer standards apparently proved to be the chief obstacle to the success of the scheme.

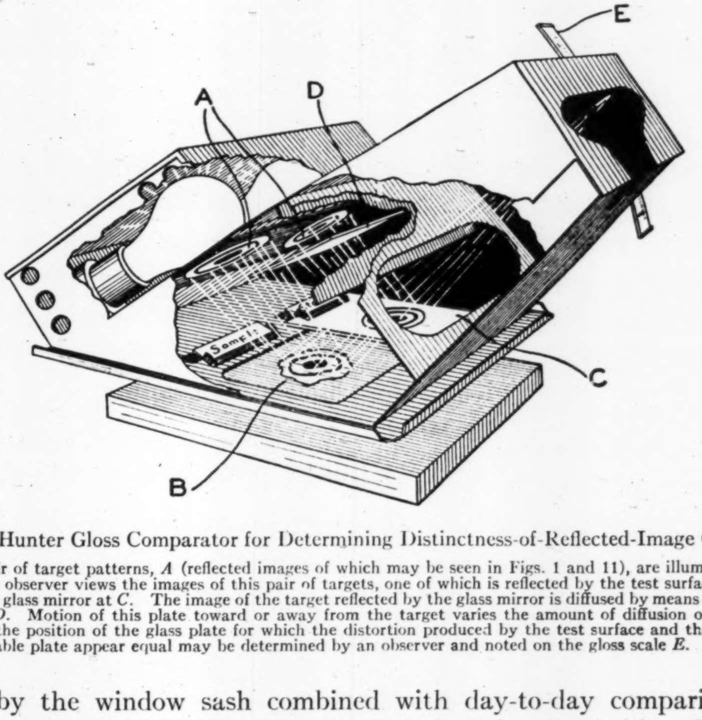

FIG. 13.—Hunter Gloss Comparator for Determining Distinctness-of-Reflected-Image Gloss (18).

A pair of target patterns, *A* (reflected images of which may be seen in Figs. 1 and 11), are illuminated from behind. An observer views the images of this pair of targets, one of which is reflected by the test surface at *B*, the other by the glass mirror at *C*. The image of the target reflected by the glass mirror is diffused by means of a ground-glass plate *D*. Motion of this plate toward or away from the target varies the amount of diffusion of the target image, and the position of the glass plate for which the distortion produced by the test surface and that produced by the movable plate appear equal may be determined by an observer and noted on the gloss scale *E*.

The Gloss Comparator developed by the author (18), Fig. 13, is an instrument designed to measure distinctness-of-reflected-image gloss on a continuous scale.

ABSENCE-OF-SURFACE-TEXTURE GLOSS

The term, surface texture, is used to indicate all departures from surface smoothness sufficient in magnitude to be visible to the unaided eye. It has been suggested that surface texture should not be associated with gloss because it is not a property of an elemental area of surface. However, those who customarily grade materials for gloss ordinarily rate surfaces which exhibit pimples, patterns, waviness, scratches, etc., below surfaces otherwise alike in appearance but free of texture.

Since surface texture is a function of the variation of a surface from element to element, it cannot be specified by the reflection distribution functions of a given surface test area. Photographs in specularly reflected light (designated in microscopy as "bright-field" illumination) probably furnish the best known means of recording surface texture (**18, 21**). In making such a photograph the camera must be focused upon the reflecting surface. Thus, Fig. 1 (*d*), is a photograph showing the comparison of images reflected by two surfaces, one designated as "pimpled," the other as "orange peel." Inasmuch as these two surfaces both produced very distinct images when the camera was properly focused on them, the two images observed here demonstrate that a camera focused upon the glossy surface to record texture clearly does not at the same time focus on the image of the object reflected by the surface.

Many phenomena interesting psychologically are associated with surface texture. There are rivalries for visual fixation between the texture of the surface and the images reflected in the surface. Then, stereoscopic effects may be present because of the fact that an observer's two eyes receive images from a single source reflected in two different areas of the image-reflecting surface, with the result that the appearance to one eye is often quite different from the appearance of the same object to the other eye.

In the earlier paper by the author (**18**) it was suggested that the characteristic of appearance which is commonly known as "depth of finish" might be explained as being due to lack of surface texture. Apparently an object possesses "depth of finish" when the surface of this object presents no texture upon which the one who views the surface may fixate. When the surface of an object is thus nearly invisible, the observer tends to see details of grain or mottling as appearing to be at an unlocated depth within the object.

SUMMARY

The problems of gloss determination differ from many other problems of measurement in that the principal problem seems to be not how to measure the quantities involved, but rather how to determine the best quantities to measure. Gloss is associated with the ability of objects to reflect light specularly. The structural properties of materials responsible for specular reflection and the glossiness of these materials which results from specular reflection can be described at some length, but the specular reflectance, itself, with most materials cannot be measured because it cannot be separated in any but an approximate way from the diffuse reflectance. Reflection distribution functions, though complex and cumbersome, offer the only means by which the reflectance properties of surfaces responsible for their glossiness may be completely specified.

Different types of gloss may be classified according to their appearance characteristics. Six types are identified in the present paper as follows: (1) specular gloss, identified by surface shininess; (2) sheen, identified by surface shininess at grazing angles; (3) contrast gloss, identified by contrasts between specularly reflecting areas of surfaces and other areas; (4) absence-of-bloom gloss, identified by the absence of excess reflection (haze or smear) adjacent to reflected high lights; (5) distinctness-of-reflected-image gloss identified by the distinctness of images reflected in surfaces, and (6) absence-of-surface-texture gloss, identified by the lack of surface texture and points of fixation which locate the surface. If the designs of existing glossmeters are examined they will usually be found to measure one or more of the above types of gloss. The primary requirement for each of these glossmeters is that, when applied to specimens of the type for which it is intended, it shall give results which correlate satisfactorily with gloss ratings on the same specimens assigned by visual grading. In order to describe glossmeters so that the measurements they make may be reproduced, it is necessary to specify precisely the apparent-reflectance measurement made. Each type of gloss is considered separately and the devices by which each may be measured described. Goniophotometric data provide a basis for the design and improvement of gloss-determining devices.

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APPLICATIONS OF THE WEDGE EXTENSOMETER

By Wilbur M. Wilson[1]

Synopsis

The wedge extensometer, as its name suggests, is an extensometer in which the properties of the wedge are used as a means of multiplying a very small change in distance between two points, thereby making possible a more accurate measurement of the change. The instrument when used by a skilled operator is both reliable and sensitive. It is limited in its field of application but, fortunately, can be used in situations to which other extensometers are not adaptable. This paper consists of a description of several adaptations of the instrument as used by the author and by assistants working under his direction.

The first use by the author of a wedge as a means of measuring a change in the distance between two points was in connection with static tests to determine the bearing value of large cylinders.[2] One of the objects of this investigation was to determine the relation between the load and the area in contact for a loaded cylinder bearing on the plane top of a base. The cylinders (actually segments of cylinders were used) varied in diameter from 40 in. to 480 in. The method of making a test was to apply a load on the cylinder, insert two steel shims 0.003 in. thick between the cylinder and the base, one on each side of the area in contact, and measure the distance between the shims by means of a wedge, as indicated in Fig. 1. The wedge used for most of these tests had a taper of 1 to 10 so that the vertical movement of the wedge was ten times as great as the change in the horizontal distance between the shims. Extreme sensitiveness was not required and the vertical position of the wedge relative to the shims was determined from the scales along the two edges of the wedge. The points on Fig. 2, which is a record of an actual test, fall on a smooth curve, indicating that the instrument was sufficiently accurate for the tests for which it was used. This is the first and the most elementary of the many applications of the wedge as a means of measuring increments of distance that have been made by the author.

The wedge proved to be so satisfactory as a means of measuring the distance between the shims, in the tests to determine the bearing value of large cylinders, that it was also used in tests to determine the bearing value of small rollers[3] for which a much greater precision was necessary.

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[1] Research Professor of Structural Engineering, University of Illinois, Urbana, Ill.

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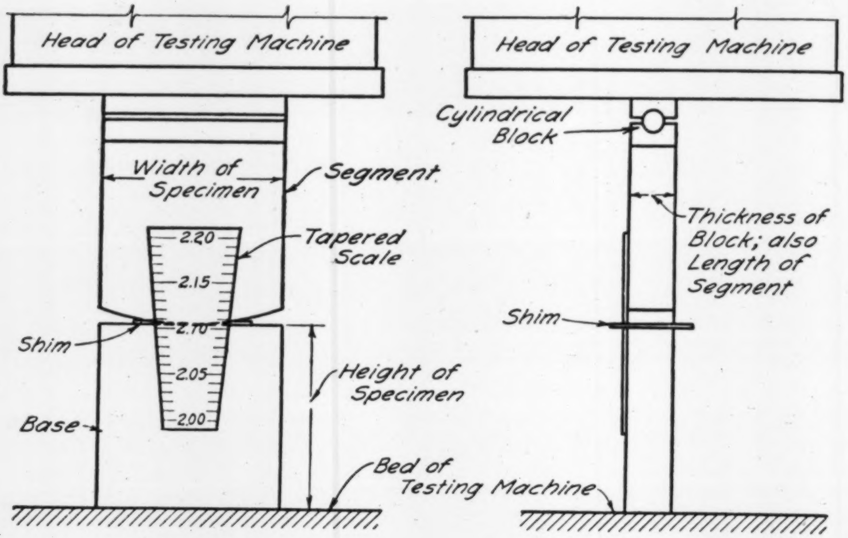
Head of Testing Machine
Width of Specimen
Segment
Tapered Scale
2.20
2.15
2.10
2.05
2.00
Shim
Base
Height of Specimen
Bed of Testing Machine
Head of Testing Machine
Cylindrical Block
Thickness of Block; also Length of Segment
Shim

FIG. 1.—Specimens for Static Tests of Segments.

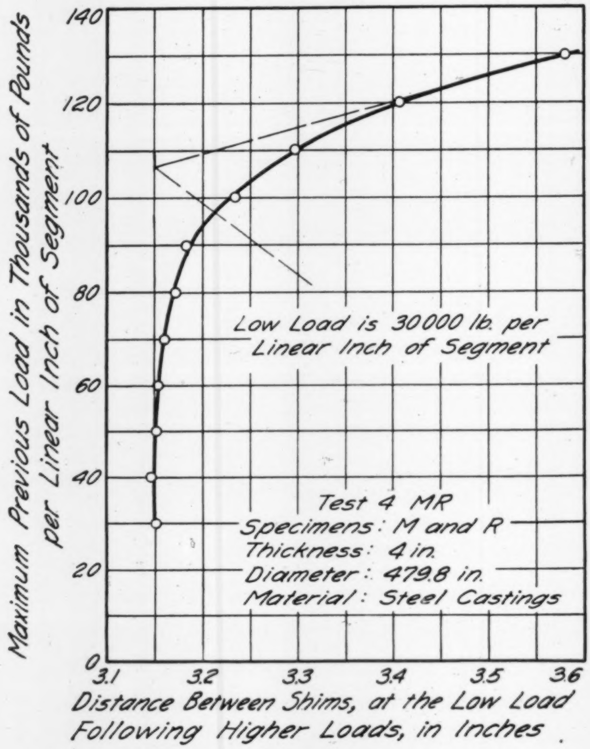

FIG. 2.—Load that Produces Set.

In these tests, a loaded roller was supported on the plane horizontal top surface of a bearing block. The object of the test was to determine the relation betwen the load and the vertical deformation and also the relation between the load and the vertical set of the material adjacent to the areas in contact. The rollers were of small diameter and the intensity of the vertical pressure decreased very rapidly with the vertical distance from the surfaces in contact and nearly all of the elastic vertical deformation and probably all of the vertical set occurred in a space not more than 1.0 in. high. A high degree of precision was therefore required. A carefully prepared steel wedge was used and its longitudinal position was determined with an Ames dial so located that the end of the wedge bore upon the plunger of the dial. Figure 3 shows the instrument attached to a roller about 6 in. in diameter. In this figure, *A* is the wedge and *B* and *C* are the support for the Ames dial. The wedge is held in position by three pins, two below the wedge and projecting from the face of the roller, and one above the wedge and projecting from the face of the base. The wedge is removed while the load is changed. Vertical movement between the single pin in the base and the two pins in the roller is indicated by the distance to which the wedge can be inserted between the pins. Pins and dials were provided on both the near and the far sides of the specimen, but the one wedge was used for the two sets of readings. The pins were made of drill rod and the wedge was made of tool steel, hardened, ground and finished to a smooth plane surface. It had a taper of 50 to 1 so that

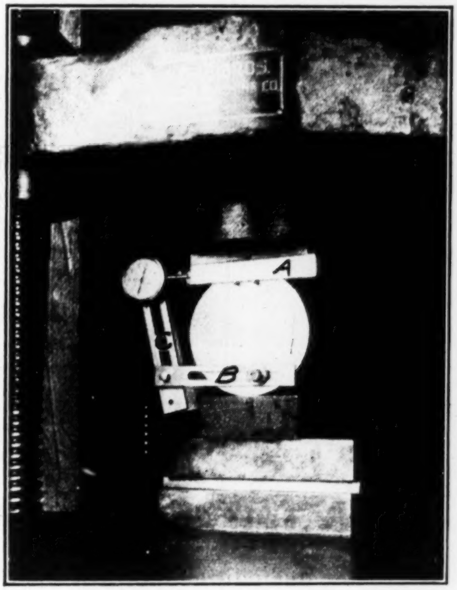

FIG. 3.—Tests of Small Rollers.

one division of the dial indicated a vertical deformation of 0.00002 in. Readings were taken in duplicate, the wedge being removed from the pins between readings, and no difficulty was experienced in working to a tolerance of one division.

A test similar to the one described in the previous paragraph was used to determine the bearing value of pivots for scales.[4] A loaded V-shaped pivot bore upon the plane horizontal top surface of a base. The test was to determine the relation between the load and the vertical deformation, and also the relation between the load and the vertical set. The vertical deformation is even more localized for the pivots than for the small rollers. The wedge extensometer used in these tests differed from the one shown in Fig. 3 in this respect: the restraint for the wedge, instead of being pins projecting from the face of the specimens, consisted of auxiliary knife-edges embedded in steel bars, one pair clamped to the pivot and the other to the base. The instrument gave excellent results as indicated by the fact that these results plotted along smooth curves.

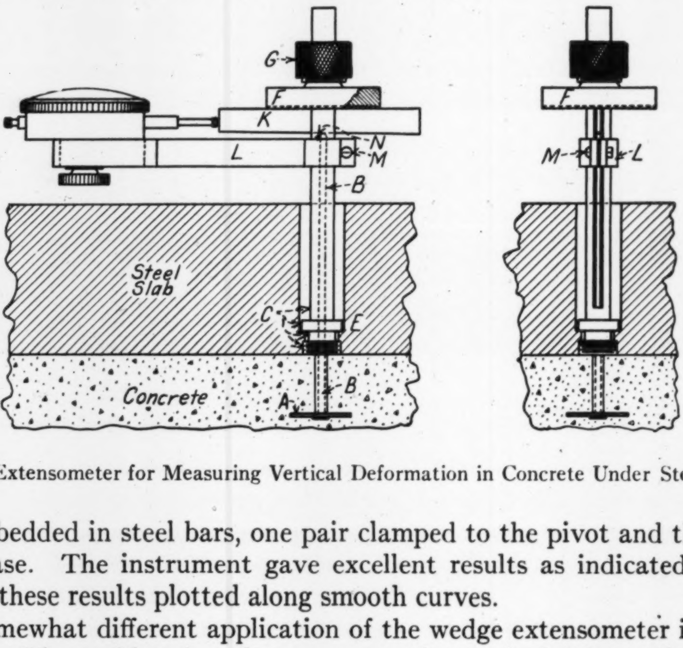

Fig. 4.—Extensometer for Measuring Vertical Deformation in Concrete Under Steel Slab.

A somewhat different application of the wedge extensometer is shown in Fig. 4. The problem here is to measure the vertical deformation of the concrete at various points under a loaded steel slab carrying a concentrated load. The $\frac{1}{2}$-in. rod *C* is attached to the steel slab at its lower end so that it moves vertically with the lower surface of the slab. It is bored to receive the $\frac{1}{8}$-in. drill rod *B*, attached rigidly to the thin disk *A*, which is embedded in the concrete. The lower portion of *B* is encased in a soft rubber tube so that its vertical motion is not influenced by the concrete along its length but determined entirely by the vertical motion of the disk, which moves with the concrete immediately adjacent to it. Thus the vertical movement of *B* relative to *C* is the vertical deformation of the concrete between the disk *A* and the lower side of the steel slab. The disk *F*, made of hardened tool steel, has a thin projecting edge on the lower side as shown in the figure and is provided with a clamp and nut *G* which will hold it firmly in any desired position along the rod *C*. The upper portion of *C* is slotted to receive the wedge *K*. An Ames dial is supported on the bracket *L* clamped to the rod *C* in such a manner that it can be moved up or down on the latter to any desired position. The pin *N* extends through *C* and is a backing-up support to hold the upper end of rod *B* in a fixed position as the wedge *K* is pushed through the slot over it. In the preliminary adjustment of the instrument with no load upon the slab, the wedge is entered into the slot until the dial plunger is moved nearly its entire range. The disk *F* is then pushed down until its edges are in contact with the top edge of the wedge, and clamped in position. The bracket *L* is then adjusted for position to bring the plunger of the dial in the desired position relative to the end of the wedge. The three points of contact on the wedge are now the two edges of disk *F*, on top of the wedge, and the rounded upper end of the rod *B*. Any vertical motion of *B* relative to *C* will vary the distance which the wedge can enter between these three points of support, a distance indicated by the reading of the dial.

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[4] Wilbur M. Wilson, Ray L. Moore and Frank P. Thomas, "Bearing Value of Pivots for Scales," *Bulletin No. 242*, University of Illinois Engineering Experiment Station (1932).

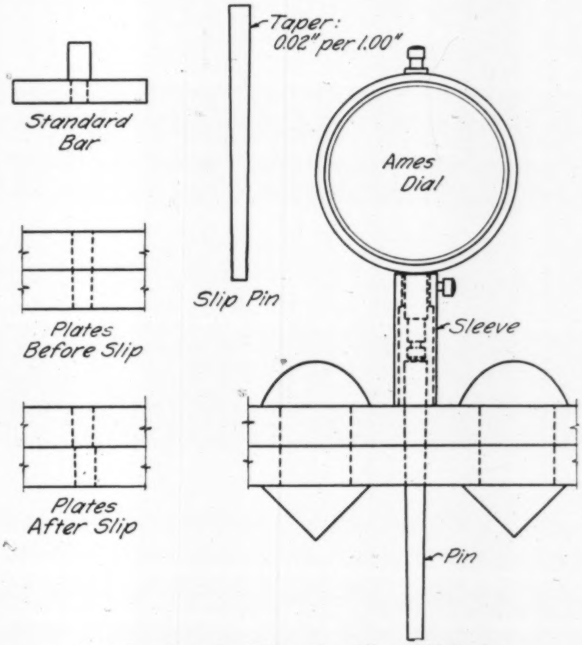

FIG. 5.—Gage for Measuring Slip, 1930 Series.

Mr. Holder, by having a number of these instruments attached to a slab, was able to measure satisfactorily the vertical deformation at various points over the top 1 in. of the concrete directly under the slab when the latter was subjected to a single concentrated load.

The tapered pin as a means of measuring the slip between adjacent plates of a riveted joint is a very successful adaptation of the wedge extensometer in which the tapered pin replaces the flat wedge used in the other instruments. The tapered slip pin was first used by the author in tests of joints in wide plates.[5] The scheme of the instrument is apparent from Fig. 5. A hole drilled through the plates is reamed to the same taper as the pin to be used. The distance the pin can be entered is measured by means of the Ames dial mounted as indicated in the figure. A standard bar is used to check the instrument against accidental changes in adjustment. When the plates slip relative to each other, one diameter of the clear hole is reduced, thereby decreasing the distance that the pin can be entered, the change in this distance being the product of the slip and the taper of the pin. Standard tapered pins and similar tapered reamers having a taper of 0.25 in. per ft. (a nominal taper of 1 to 50), which can be purchased for a few cents, prove entirely satisfactory. This instrument has fulfilled a real need for a reliable and sensitive instrument that would measure small amounts of slip, one for which the preparation of the specimen is inexpensive and does not seriously affect the strength of the plate. If the joint is of such a character that one plate will bend relative to the other, the hole in one plate should be enlarged from the outer surface to within about $\frac{1}{16}$ in. of the inner surface; otherwise the reduction in the distance the pin will enter the hole may be due to a change in direction of the axis of the hole in one plate relative to that in the other instead of being due to the slip of one plate relative to the other.

Acknowledgments:

The assistants who helped develop the instruments and who made the tests reported in the various bulletins referred to were, at the time the work was done, half-time graduate research assistants in Civil Engineering at the University of Illinois working under the supervision of the author, and are as follows: Tests of the bearing value of large rollers, George M. Keranen, James Hardesty, W. K. Brown, and E. C. Hartmann; the bearing value of rollers, Alfred Hedefine; bearing value of pivots for scales, Frank P. Thomas; and test of joints in wide plates, James Mather and Charles O. Harris. The instrument for measuring the vertical deformation of the concrete beneath a loaded slab was developed and used by Paul Davie Holder, Ph.D., M.Sc., while a graduate student in Civil Engineering working under the supervision of the author.

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[5] Wilbur M. Wilson, James Mather and Charles O. Harris, "Tests of Joints in Wide Plates," *Bulletin 239*, University of Illinois Engineering Experiment Station (1931).

DISCUSSION

Mr. N. B. Pilling[1] (*presented in written form*).—I should like to confirm Mr. Wilson's remarks about the usefulness of the wedge principle in extensometers. Our laboratory has had occasion to deal with slippage in riveted joints and after consideration of the difficulties attending the measurement of relative motion with the conventional types of gage-length extensometers, adopted the wedge principle with most satisfactory

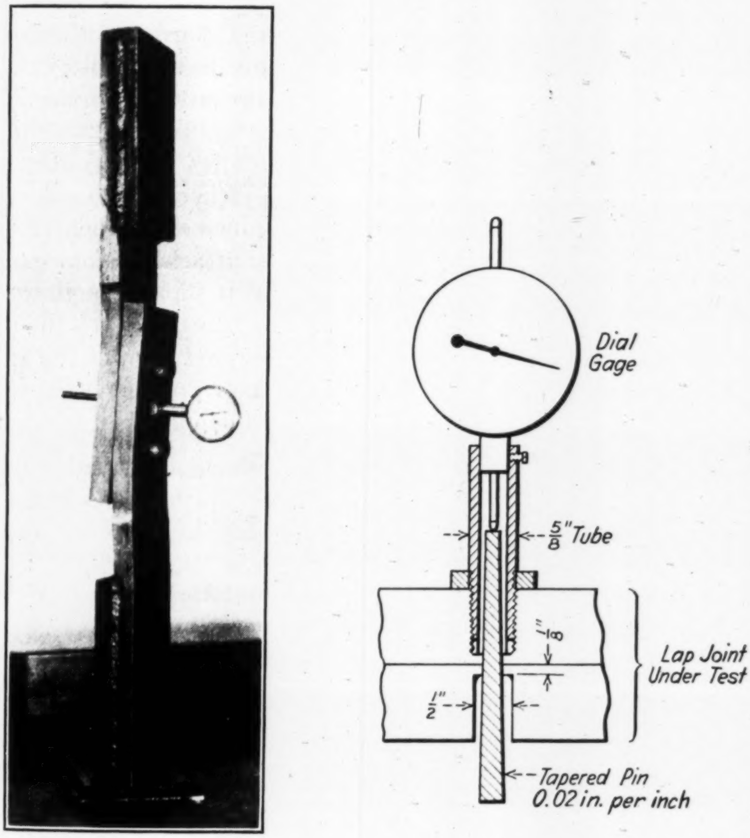
Dial Gage
5/8" Tube
1/8"
Lap Joint Under Test
1/2"
Tapered Pin 0.02 in. per inch

Fig. 1.—Wedge Extensometer.

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[1] Manager, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

results. In this type of application the wedge extensometer combines compactness and ruggedness with sensitivity.

Referring to Fig. 5 of the paper, each reading is made by seating the tapered pin and then applying the dial to the protruding pin. We have made a different arrangement which improves the convenience of operation by eliminating handling of the sensitive dial gage. This consists in screwing the gage permanently to the test plate with a threaded tube and reversing the position of the pin relative to the dial. In this way only the tapered pin is handled and direct dial readings are obtained. The accompanying Fig. 1 shows this in detail.

MR. F. G. TATNALL.[2]—One additional application of the wedge extensometer was first announced by Kenyon and Burns, of the American Rolling Mill Co., in 1933. They used the wedge in an ordinary separator extensometer for use on sheet metal. It was operated in connection with a recording drum such as is found on any universal testing machine which has facilities for taking a load record. It measures the specimen elongation up to and through the breaking point at a ratio of ten to one. As the specimen extends, the wedge slips through the increasing opening between the two separate parts of the extensometer, one attached to one gage point and the other to the second gage point, and as it slips through under the action of a weight, it pulls the pencil across the chart or turns the drum as the case may be and records very faithfully the extension of the specimen on the chart. This form of extensometer is now commercially available and is coming into wide usage in sheet metal testing.

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[2] Manager, Testing Equipment Dept., Baldwin-Southwark Corp., Philadelphia, Pa.

EDGE CORRECTION IN THE DETERMINATION OF DIELECTRIC CONSTANT[1]

By Harvey L. Curtis[2] and Arnold H. Scott[2]

Synopsis

An edge correction is often required in determining the dielectric constant of an insulating material by the capacitance method. The dielectric constant is generally defined as the ratio of the capacitance between two electrodes completely surrounded by the material to the capacitance between them when the material is removed. An edge correction is required when the dielectric does not completely surround the electrodes or when the material cannot be removed without disturbing them so that the capacitance with a vacuum surrounding the electrodes must be computed from their dimensions. An edge correction is particularly important in determining the dielectric constant of a material in the form of sheets, provided it is not feasible to employ a guard ring.

The edge correction is discussed for ten different arrangements of electrodes, all in the form of plates. Six of these are concerned with circular electrodes, the remainder with narrow strips. The error in the dielectric constant which may arise from the use of the formulas is considered for each of the different arrangements. It is shown that some arrangements give a value that is too large; others, one that is too small. It is pointed out that one arrangement is superior to the others.

An edge correction is usually required in those capacitance methods of determining dielectric constant that employ only two electrodes. This necessity arises from the difficulty of making the experimental conditions of a method conform to the requirements implied in the definition of dielectric constant. The dielectric constant of a material is defined as the ratio of the capacitance of a capacitor having the material as a dielectric to the capacitance of another capacitor which has an identical arrangement of electrodes, but has a vacuum as the dielectric. This may conveniently be represented by the equation

$$K = \frac{C_k}{C_v} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

where K = the dielectric constant of the material,

C_k = the capacitance of a capacitor with the material as its dielectric, and

C_v = the capacitance of a capacitor having an identical arrangement of electrodes as for C_k but having a vacuum[3] as its dielectric.

This definition assumes that, for each capacitor, the dielectric completely fills the electric field surrounding the electrodes. The edge correction is a modification that can be applied to Eq. 1 to allow for the fact that the electric field is not confined to the space immediately between the electrodes.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Principal Physicist, and Assistant Physicist, respectively, National Bureau of Standards, Washington, D. C.

[3] Except for measurements of high precision, air, having a dielectric constant of 1.00059 under normal conditions, may be used instead of a vacuum.

The principal application of the edge correction is in determining the dielectric constant of solids in the form of sheets. A capacitor is constructed from a sheet of the material by placing an electrode on each side. The capacitance of this capacitor is measured and the capacitance of a capacitor which is identical, except that it has vacuum as a dielectric, is computed. The edge correction is introduced to correct the measured capacitance for the fact that the dielectric under investigation does not completely fill the electric field surrounding the electrodes and to give an accurate formula for determining the computed capacitance. To avoid the necessity of applying an edge correction, a capacitor having a guard electrode is often employed. Such a capacitor is not discussed in this paper.

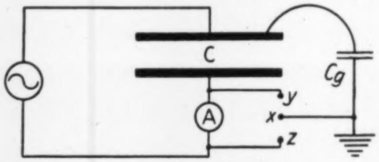

FIG. 1.—Diagram Showing the Effect on the Measured Capacitance of the Point at Which the Ground Connection Is Made.

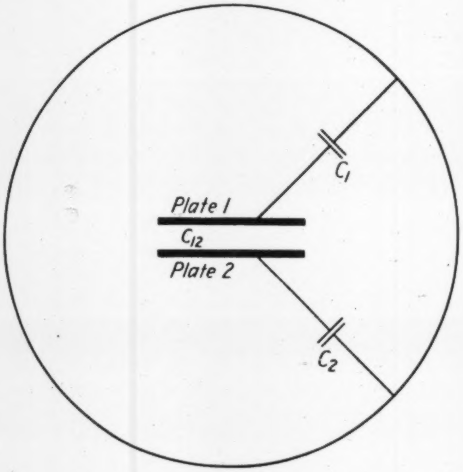

FIG. 2.—Diagram Illustrating the Coefficients of Capacitance of Two Parallel Plates Surrounded by an Infinite Sphere.

Ten different arrangements of electrodes will be considered. Six arrangements apply to a single sheet of material with electrodes on the opposite sides. Four other arrangements apply to a pair of sheets with one electrode between them and with their outer electrodes joined to form a single electrode.

A SINGLE SHEET OF DIELECTRIC

A sheet of dielectric material is prepared for determination of its dielectric constant by placing electrodes on its two opposite faces. This constitutes a capacitor, the capacitance of which can be measured by comparison with a standard capacitor. However, the measured capacitance may include the capacitance between one or both electrodes and the earth. The contribution of this earth capacitance depends on the connection to earth of the measuring circuit. Even with one electrode at earth potential,

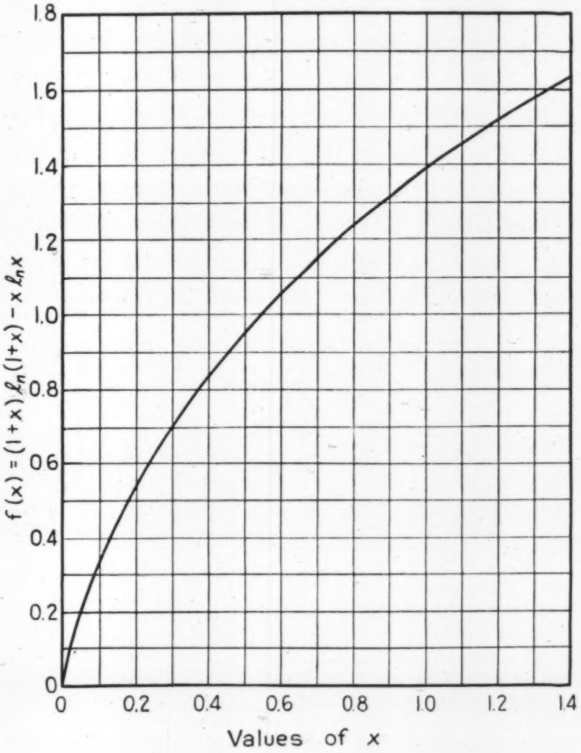

FIG. 3.—Values of a Function Used in Computing the Edge Capacitance.

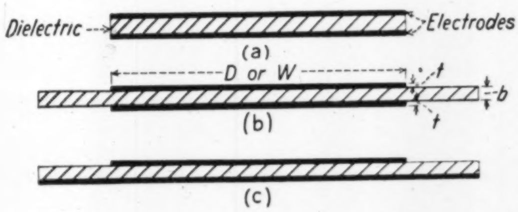

FIG. 4.—Arrangement of Electrodes on a Single Sheet of Dielectric.

a change in the connection to earth may change the measured capacitance. This is illustrated by the diagram of Fig. 1. The measured capacitance is determined from the value and frequency of the potential difference between the plates of the capacitor and the value of the current in the circuit as measured by the ammeter A. If this ammeter has a negligible impedance relative to that of the capacitor, the lower plate of the capacitor will be at earth potential when the earth connection is made on either side of the ammeter. If the connection is below the ammeter (x connected to z), the measured current is that through C only, while if the connection is above the ammeter (x connected to y), the measured current is that through both C and C_g. In the following discussion, the measuring circuit is always so arranged that only the capacitance C is measured. The method of making the electrical measurements will not be considered, so that the manner of attaching the leads and determining their capacitance will not be discussed.

While the experimental arrangement is assumed to be such that only the capacitance between the electrodes is measured, yet the theoretical discussion will be simplified by considering the system as two conducting plates situated near the center of a conducting sphere of infinite radius. If the plates are circular disks that are coaxial, and if the sphere is evacuated, all the coefficients of capacitance can be computed from the dimensions of the disks. If the plates are long rectangles, no formula is available for computing the capacitance to earth.

The arrangement of electrodes is shown diagrammatically in Fig. 2. The mutual capacitance between plates 1 and 2 is designated as C_{12}, while the capacitances between the plates and the infinite sphere are designated as C_1 and C_2. It is convenient to consider C_{12} as made up of two parts, C_n and C_e. The normal capacitance, C_n, is the value that C_{12} would have if the electricity were uniformly distributed over the inner surfaces of the electrodes and if the lines of electrostatic intensity were all straight lines extending directly from one electrode to the other. The edge capacitance, C_e, is the correction that must be added to C_n to give C_{12}. Values of C_n and C_e can be computed for circular electrodes and C_n and $(C_e + C_1)$ for long, rectangular electrodes.

Circular Electrodes:

The formulas for computing the above capacitance[4] for circular electrodes are:

$$C_n = \frac{1.113\,D^2}{16b} \text{ micromicrofarads} \ldots\ldots\ldots\ldots (2)$$

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[4] G. Kirchhoff, "On the Theory of Condensers," *Monatsbericht* der Akademie der Wissenschaften zu Berlin, March, 1877, p. 144; also *Collected Works*, p. 101, Barth, Leipzig (1882). A more recent paper is by J. W. Nicholson, *Philosophical Transactions*, Vol. 224A, p. 303 (1924). Nicholson makes an exact solution, but his formula contains unsolved integrals.

$$C_e = \frac{1.113\,D}{8\pi}\left[ln\,\frac{8\pi D}{b} - 3 + z\right] \text{ micromicrofarads} \ldots\ldots\ldots (3)$$

$$C_g = C_1 = C_2 = \frac{1.113\,D}{2\pi} \text{ micromicrofarads} \ldots\ldots\ldots (4)$$

where D = diameter of each electrode in centimeters,
b = distance between the inner faces of the electrodes in centimeters,
t = thickness of each electrode in centimeters, and

$$z = (1 + x)\,ln\,(1 + x) - x\,ln\,x \ldots\ldots\ldots (5)$$

in which $x = t/b$, and
ln = signifies a natural logarithm (2.303 times the common logarithm).

Values of z for any given value of x can be obtained from the curve of Fig. 3. From this curve it is seen that for any value of x less than 0.1 (t less than $b/10$), the value of z is given with sufficient accuracy by the expression

$$z = 4x = \frac{4t}{b} \ldots\ldots\ldots (6)$$

Hence, for thin electrodes

$$C_e = \frac{1.113\,D}{8\pi}\left[ln\,\frac{8\pi D}{b} - 3 + \frac{4t}{b}\right] \text{ if } t < b/10 \ldots\ldots\ldots (7)$$

The number 1.113 is the factor required to convert c.g.s. electrostatic units of capacitance into micromicrofarads. The expression for C_g is given for the convenience of those who may wish to modify the methods herein outlined. These formulas can be applied to three arrangements of electrodes, all of which are frequently employed in measuring dielectric constants.

A Circular Sheet of Dielectric Having Electrodes of the Same Diameter as the Sheet.—A capacitor consisting of a circular sheet of dielectric with electrodes completely covering both surfaces, as shown in Fig. 4(a), has a capacitance, C, when measured as previously described with one electrode at earth potential, which is approximately given by the equation:

$$C = KC_n + C_e \ldots\ldots\ldots (8)$$

where K is the dielectric constant of the material, so that

$$K = \frac{C - C_e}{C_n} \ldots\ldots\ldots (9)$$

$$= \frac{16\,bC}{1.113\,D^2} - \frac{2b}{\pi D}\left[ln\,\frac{8\pi D}{b} - 3 + z\right] \ldots\ldots\ldots (10)$$

In this arrangement of electrodes, the edge capacitance is probably larger than C_e because the configuration of the electric field at the edge is slightly different when the dielectric is between the electrodes from that when vacuum is the only dielectric. Hence, the value of K obtained by this method may be larger than the true value.

The accuracy of this method was tested by Büchner.[5] He tested potassium chloride (dielectric constant 4.68) using sheets of material of various diameters and thicknesses. The maximum variation of any value of the dielectric constant from the mean was 0.03 (0.7 per cent).

A Sheet of Dielectric Extending Some Distance Beyond the Edges of the Circular Electrodes that Are Coaxial and Have the Same Diameter.—A capacitor which consists of a relatively large sheet of dielectric between two equal circular electrodes that have their centers directly opposite each other, as shown in Fig. 4(b), has a measured capacitance, C, which is approximately given by the equation:

$$C = K(C_n + C_e) \qquad \text{(11)}$$

Hence,

$$K = \frac{C}{C_n + C_e} \qquad \text{(12)}$$

$$= \frac{C}{1.113\left\{\dfrac{D^2}{16b} + \dfrac{D}{8\pi}\left[ln\,\dfrac{8\pi D}{b} - 3 + z\right]\right\}} \qquad \text{(13)}$$

In these formulas, $K\,C_e$ is probably too large, because the lines of electric intensity at the edge do not all pass from one electrode to the other entirely in the dielectric. Hence, the value of K obtained by this method may be smaller than the true value.

The accuracy of this method was tested by Grüneisen and Giebe.[6] They tested porcelain (dielectric constant 5.60) using sheets of various thicknesses. The maximum variation of any value of the dielectric constant from the mean was 0.04 (0.7 per cent). They conclude, however, from measurements on capacitors made of two sheets of porcelain, that the dielectric constant obtained by this method is too small by about 4 per cent.

Hoch[7] experimentally determined the edge correction for this method using rubber (dielectric constant about 3) of three different thicknesses. He found that the edge capacitance was about 35 per cent less than $K\,C_e$.

The data obtained by these experimentalists show that the dielectric constant obtained by this method is too small, as was predicted from theoretical considerations, but the amount depends on the diameter and thickness of the electrodes and on the diameter, thickness, and dielectric constant of the sheet of dielectric.

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[5] A. Büchner, "Measurement of Dielectric Constants," *Zeitschrift für Technischen Physik*, Vol. 16, p. 10 (1935).

[6] E. Grüneisen and E. Giebe, "Use of the Three-Plate Capacitor for the Determination of the Dielectric Constant of Solid Bodies," *Verhandlungen* der Deutschen Physikalischen Gesellschaft, Vol. 14, p. 921 (1912).

[7] E. T. Hoch, "Electrode Effects in Measurement of Power Factor and Dielectric Constant of Sheet Insulating Materials," *Bell System Technical Journal*, Vol. 5, p. 555 (1926).

A Sheet of Dielectric Having One Large Electrode and One Small, Circular Electrode.—A capacitor consisting of a sheet of dielectric between a large electrode which nearly or completely covers one face, and a small circular electrode near the center of the opposite face, as shown in Fig. 4(*c*), has a capacitance, when measured in the manner already described with the large electrode at earth potential, which is approximately given by the equation:

$$C = K(C_n + C'_e) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (14)$$

where C_e' is twice the value of the edge capacitance of an equal electrode capacitor in which the electrodes have the same diameter as the smaller electrode and the distance between the electrodes is twice the actual distance. Hence,

$$K = \frac{C}{C_n + C'_e} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (15)$$

$$= \frac{C}{1.113\left\{\frac{D^2}{16b} + \frac{D}{4\pi}\left[ln\,\frac{4\pi D}{b} - 3 + z_3\right]\right\}} \quad \ldots\ldots\ldots (16)$$

where D = diameter of the smaller electrode in centimeters,
b = thickness of the dielectric in centimeters, and
t = thickness of smaller electrode in centimeters (required in evaluating z_3).

In determining z_3 from Eq. 5 or Fig. 3, $x = t/2b$.

In the above arrangement of electrodes, $K\ C_e'$ is probably too large, because some of the lines of electric intensity at the edge pass partly through air. Hence, the value of K obtained by this method may be smaller than the true value.

While the formula for this arrangement of electrodes follows directly from Kirchhoff's formula, it is not in general use. Hence, no experimental confirmation is available.

Long Rectangular Electrodes:

The normal capacitance C_n and the sum of the edge capacitance C_e and the earth capacitance C_g, when the electrodes are long rectangular plates which form the opposite faces of a rectangular parallelopiped and when they are in an evacuated infinite sphere, are given by the following formulas:

$$C_n = \frac{1.113\, lw}{4\pi b} \text{ micromicrofarads} \ldots\ldots\ldots\ldots\ldots (17)$$

$$C_e + C_g = 1.113\, \frac{l}{4\pi^2}\left\{1 + ln\left[1 + \frac{\pi w}{b} + ln\left(1 + \frac{\pi w}{b}\right)\right]\right\} \text{ micromicrofarads..} (18)$$

where b = the thickness of the dielectric in centimeters,
w = width of the electrodes in centimeters, and
l = length of the electrode in centimeters.

No formula is available for separating C_e and C_g. In the following discussion on rectangular electrodes the assumption is made that C_g is so small that it can be neglected.

The formulas were deduced from the equation given by Thompson[8] for the distribution of electricity along the edges of two semi-infinite, parallel electrodes which have their edges in a plane perpendicular to the electrodes. Coursey[9] gives a formula for the capacitance of rectangular plates which is the sum of Eqs. 17 and 18. Equation 18 assumes that the strip electrodes are so wide that the effect of one edge does not extend as far as the opposite edge. Furthermore, the derivation does not consider the thickness of the electrodes nor the correction at their ends. Hence, this formula should be used only when the electrodes are thin and wide relative to the thickness of the dielectric and when their length is several times their width. Even with these restrictions, the formula for C_e is probably less accurate than the similar formula with circular electrodes, especially since C_g is included. However, there is no experimental evidence to substantiate this statement. Three different arrangements of electrodes will be considered that are analogous to the three cases for circular electrodes.

A Long Sheet of Dielectric with Electrodes Covering Both Faces.—Any capacitor consisting of a sheet of dielectric with electrodes that cover both faces, as shown in Fig. 4(*a*), can be represented by Eq. 8, and the dielectric constant determined by Eq. 9, provided C_n and C_e can be computed. Formulas for computing C_n and C_e for long electrodes are given above. However, the dielectric constant, when determined with long, narrow electrodes, is probably less accurate than when determined with circular electrodes.

A Sheet of Dielectric with Long, Narrow Electrodes.—A capacitor that consists of a dielectric that projects beyond the edges of the long, narrow electrodes, which are so placed on the faces of the dielectric that they are opposite faces of a rectangular parallelopiped, as shown in Fig. 4(*b*), has a measured capacitance that is given by Eq. 11, provided C_n and C_e are computed by Eqs. 17 and 18. The dielectric constant can then be computed by Eq. 12, but the result will probably be less accurate than for the similar case using circular electrodes.

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[8] J. J. Thompson, "Recent Researches in Electricity and Magnetism," p. 216, Clarendon Press, Oxford, England (1893).

[9] P. R. Coursey, "Electrical Condensers," p 138, Sir Isaac Pitman and Sons, Ltd., London (1927).

A Sheet of Dielectric with Long Electrodes, One of Which Is Narrower than the Other.—A capacitor that consists of a long sheet of dielectric, with an electrode nearly or completely covering one face, and a long, narrow electrode on the opposite face, as shown in Fig. 4(*c*), has a capacitance given by Eq. 14. The value of C'_e for one electrode narrower than the other can be obtained from C_e in the same manner as was done for the analogous case with circular electrodes. Hence,

$$C'_e = 1.113\,\frac{l}{2\pi^2}\left\{1 + ln\left[1 + \frac{\pi w}{2b} + ln\left(1 + \frac{\pi w}{2b}\right)\right]\right\} \text{ micromicrofarads} \ldots (19)$$

Then the dielectric constant can be computed by Eq. 15. The uncertainty in the formula on which this is based does not inspire confidence in the result. There are no experimental data which confirm or refute it.

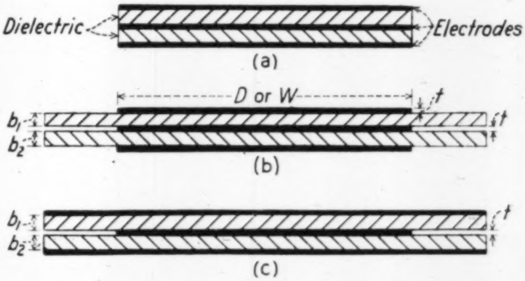

FIG. 5.—Arrangement of Electrodes when Two Sheets of Dielectric Are Used.

TWO SIMILAR SHEETS OF DIELECTRIC

When two similar sheets of dielectric have one common electrode between them, and the two outside electrodes connected together, the edge correction of this capacitor cannot be computed by the formula given for a single sheet of dielectric. Moreover, the same symmetry does not exist between the different cases as when only a single sheet of dielectric is employed. However, the capacitance to earth does not enter in any case, and the normal capacitance, C_n, is computed by the same formula as in the preceding section.

Two Circular Sheets of the Dielectric Interleaved Between Three Conducting Disks, All Five of the Plates Having the Same Diameter.—When two circular sheets of a dielectric material, both having the same diameter, are assembled with three conducting disks having the same diameter as the sheets of dielectric, as shown in Fig. 5(*a*), the measured capacitance, C, is given approximately by the equation:

$$C = K(C_{n1} + C_{n2}) + C_e \ldots\ldots\ldots\ldots\ldots\ldots (20)$$

so that

$$K = \frac{C - C_e}{C_{n1} + C_{n2}} \quad \text{(21)}$$

where

$$C_{n1} = 1.113 \frac{D^2}{16b_1} \text{ micromicrofarads} \quad \text{(22)}$$

$$C_{n2} = 1.113 \frac{D^2}{16b_2} \text{ micromicrofarads} \quad \text{(23)}$$

$$C_e = \frac{1.113\, D}{\pi}\left[0.5539 + \frac{1}{6}(z_1 + z_2)\right] \text{ micromicrofarads} \quad \text{(24)}$$

where both z_1 and z_2 may be represented by Eq. 5.

But for z_1, $x = \frac{2\,t}{b_1}$

and for z_2, $x = \frac{2\,t}{b_2}$

D = the diameter of the electrodes in centimeters,
b_1 = the thickness of one sheet in centimeters,
b_2 = the thickness of the second sheet in centimeters, and
t = thickness of each conducting disk in centimeters.

The formula for C_e was derived by Grüneisen and Giebe,[10] who have published only the final formula and state that the derivation has not been rigorously carried through. For zero thickness of the electrodes, the formula indicates that the edge correction is independent of the thickness of the dielectric. This may be applicable only for a limited range of thicknesses and diameters. However, Grüneisen and Giebe measured and computed the capacitance of 13 air capacitors in which the diameter of the conducting disks varied from 15 to 30 cm., the distance between adjacent electrodes varied from 1 to 3 cm., and the thickness of the electrodes varied from 0.08 to 0.59 cm. The difference between the computed and observed capacitance was, in one case, 2 per cent of the measured capacitance, but in all the other twelve cases was less than 1 per cent. It appears that the difference was not greater than the error of measurement. Hence, the formula may be considered satisfactory.

The value of K obtained by this method is larger than the true value. This results from assuming that the edge capacitance, when the dielectric is between the plates, is equal to C_e. As many of the lines of electric intensity pass in part through the dielectric, the edge capacitance is somewhat larger with the dielectric than with a vacuum.

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[10] E. Grüneisen and E. Giebe, "Use of the Three-Plate Capacitor for the Determination of the Dielectric Constant of Solid Bodies," *Verhandlungen* der Deutschen Physikalischen Gesellschaft, Vol. 14, p. 921 (1912).

Two Sheets of the Dielectric Interleaved Between Three Circular, Coaxial Disks, All of the Same Diameter, but the Dielectric Extending Some Distance Beyond the Edges of the Disks.—When two large sheets of a dielectric are assembled, as shown in Fig. 5(*b*), with three circular conducting disks all of the same diameter and with their centers in a line perpendicular to the faces of the disks, the measured capacitance, C, between the center disk and the two outside ones is given by the formula

$$C = K(C_{n1} + C_{n2} + C_e) \qquad (25)$$

Hence,

$$K = \frac{C}{C_{n1} + C_{n2} + C_e} \qquad (26)$$

where the values of C_{n1}, C_{n2}, and C_e are the same as given in the preceding section.

The value of K obtained by this method is smaller than the true value. This results from assuming that the edge capacitance is equal to $K\ C_e$, the value it would have if the space around the electrodes were completely filled with the dielectric. As many of the lines of electric intensity pass in part through air, the value of the edge capacitance is less than $K\ C_e$. However, the error in K is probably not as large as with the similar method using a single sheet of dielectric. This was demonstrated by the experiments of Grüneisen and Giebe using porcelain as a dielectric. They obtained a value for the dielectric constant about 4 per cent higher with this method than with the corresponding method of the first section of this paper.

Two Sheets of Dielectric Interleaved Between Three Conducting Plates, the Center Plate Being Circular, and Having the Sheets and Other Plates Extending Some Distance Beyond Its Edge.—When two large sheets of a dielectric are assembled with three conducting plates, the center one being circular and smaller than the other plates and the sheets, as shown in Fig. 5(*c*), the measured capacitance is approximately given by the equation:

$$C = K(C_{n1} + C_{n2} + C'_e) \qquad (27)$$

so that

$$K = \frac{C}{C_{n1} + C_{n2} + C'_e} \qquad (28)$$

where

$$C_{n1} = \frac{1.113\,D^2}{16b_1} \qquad (29)$$

$$C_{n2} = \frac{1.113\,D^2}{16b_2} \qquad (30)$$

$$C'_e = \frac{1.113\,D}{\pi}\,[0.6932 + z_4 + z_5] \ldots\ldots\ldots\ldots\ldots (31)$$

where both z_4 and z_5 may be represented by Eq. 5.

But for z_4, $x = \dfrac{t}{4b_1}$

and for z_5, $x = \dfrac{t}{4b_2}$

D = diameter of the small, interior electrode in centimeters,
b_1 = thickness of one sheet of dielectric in centimeters,
b_2 = thickness of the second sheet of dielectric in centimeters, and
t = thickness of the inner electrode in centimeters.

The equation for C'_e is given by Güntherschulz[11] without any indication of its source or method of derivation. It has the same form as the equation for C_e given by Grüneisen and Giebe, and hence may have the same limitations.

Assuming that there is not appreciable error in the formula, this method gives a value of K that is more nearly correct than any of the other methods described. If the center electrode is very thin, or is embedded in the dielectric, practically all the lines of electric intensity lie entirely within the dielectric, provided the dielectric and outer electrodes extend a sufficient distance beyond the edge of the inner electrode.

Two Sheets of Dielectric Interleaved Between Three Conducting Plates, the Center Plate Being a Long, Narrow Strip of Metal and the Dielectric Sheets and Outside Plates Extending Some Distance Beyond the Edges of the Center Plate.—When two sheets of dielectric are assembled with three conducting plates, as shown in Fig. 5(*c*), so that both the dielectric sheets and outside plates extend some distance beyond the edges of the center plate which is in the form of a long strip of metal, the dielectric constant is also given by Eq. 28, but with the terms of the equation determined as follows:

$$C_{n1} = \frac{1.113\,lw}{4\pi b_1} \text{ micromicrofarads} \ldots\ldots\ldots\ldots\ldots (32)$$

$$C_{n2} = \frac{1.113\,lw}{4\pi b_2} \text{ micromicrofarads} \ldots\ldots\ldots\ldots\ldots (33)$$

$$C'_e = \frac{1.113\,l}{\pi^2}\,[1.3863 + z_4 + z_5] \text{ micromicrofarad} \ldots\ldots\ldots (34)$$

where z_4, z_5, b_1, b_2, and t are the same as above,

w = width of the center electrode in centimeters, and
l = length of the center electrode in centimeters.

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[11] A. Güntherschulz, "Handbuch der Physik," Vol. XVI, Chapter 20, Julius Springer, Berlin (1927).

The equation for C'_e was obtained from the equation given by Thompson[12] for the distribution of electricity on a semi-infinite plate with one straight edge when midway between two infinite plates. In extending this to the case here considered, the assumption is made that the center strip is so wide that the effect of one edge on the distribution of electricity does not extend to the opposite edge. Also, no correction is made for the end of the strip, so the length must be large relative to its width.

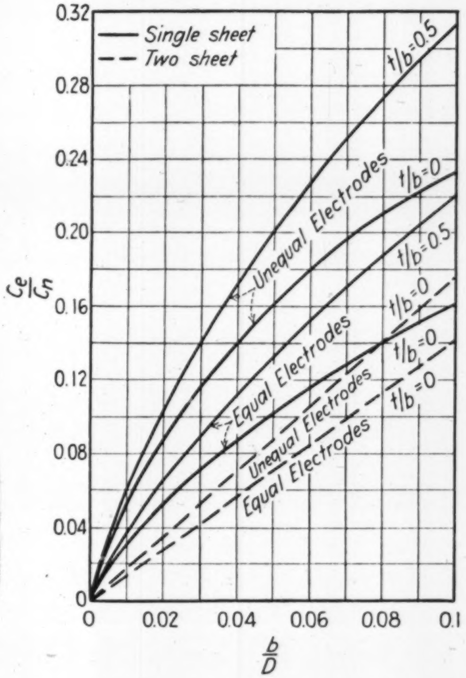

FIG. 6.—Dependence of the Edge Correction on the Relative Dimensions of the Dielectric and Electrodes when Circular Electrodes Are Used.

The accuracy attainable by this arrangement is limited because of the experimental difficulty of realizing the requirements imposed in developing the formula. There are no experimental data to indicate these limitations.

In order to indicate the magnitude of the edge capacitance with circular electrodes, the curves of Fig. 6 have been prepared. The necessary data were obtained by inserting numerical values in the formulas given for computing the capacitance when the electrodes are in a vacuum. These curves show how the edge correction depends on the dimensions and spacings of the electrodes of a vacuum capacitor for different arrangements and thicknesses of the electrodes. Only four arrangements of electrodes are required to illustrate the six cases considered for determining the dielectric constant when circular electrodes are employed. When two sheets of dielectric are employed, the center electrode is usually thin, so the curves associated with this arrangement are given only for the case where $t = 0$. When only one sheet of dielectric is employed, thick electrodes are sometimes used, so that for each arrangement of electrodes, curves are given for two relative thicknesses of electrodes.

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[12] J. J. Thompson, "Recent Researches in Electricity and Magnetism," p. 221, Clarendon Press, Oxford, England (1893).

The curves of Fig. 6 cannot be used to indicate the accuracy attainable in the value of the dielectric constant by the different methods. For example, the use of a large and a small electrode gives a larger ratio of edge capacitance to normal capacitance for a given ratio of thickness to electrode diameter than when electrodes of the same diameter are used. However, the former usually gives the more accurate value of the dielectric constant.

The edge correction for a parallel plate capacitor is important when measuring the dielectric constant of relatively thick sheets of an insulating material by any method employing a two-electrode capacitor. Some of the methods described give a value of the dielectric constant that is too large, others give a value that is too small. By collecting together these methods, it is hoped that a number of experimentalists will be encouraged to provide data for testing their reliability.

DISCUSSION

Mr. R. C. Dehmel.[1]—We have in use at the Bell Laboratories a method whereby the dielectric constant and power factor of flat or tubular specimens may be measured to frequencies of 100 kilocycles with entire elimination of edge effects. The apparatus consists of a fully shielded electrode holder (shown for circular plates in the accompanying Fig. 1) and a bridge[2] which is also shielded to permit the measurement of direct capacities and conductances. The specimen is equipped with a guard ring. These facilities may be of interest as a means for experimentally confirming the analytical expressions presented in the paper by Messrs. Curtis and Scott.

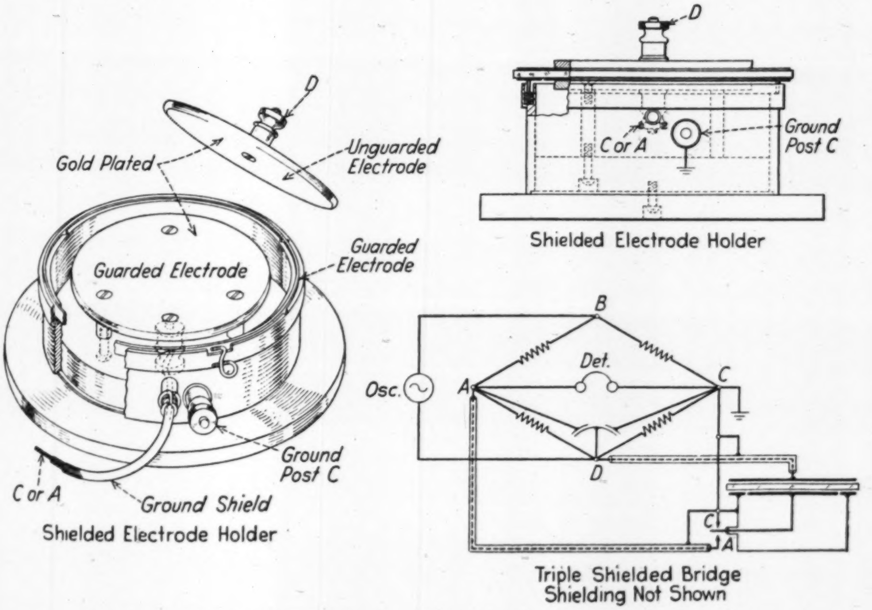
D
Gold Plated
Unguarded Electrode
Guarded Electrode
Guarded Electrode
Ground Post C
C or A
Ground Shield
Shielded Electrode Holder
D
C or A
Ground Post C
Shielded Electrode Holder
B
Det.
Osc.
A
C
D
C
A
Triple Shielded Bridge Shielding Not Shown

Fig. 1.—Apparatus for Measuring Dielectric Constant and Power Factor.

Messrs. Harvey L. Curtis[3] and Arnold H. Scott[3] (*authors' closure by letter*).—There are a number of methods and types of apparatus now in use for measuring the dielectric constants of materials when guard rings are employed. However, it was not intended that this paper should deal with this phase of dielectric constant measurements but rather with those cases where it was not feasible or possible to use guard rings.

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

[2] W. J. Shackelton and J. G. Ferguson, "Electrical Measurement of Communication Apparatus," *Bell System Technical Journal*, Vol. 7, pp. 70–89 (1928).

[3] Principal Physicist, and Assistant Physicist, respectively, National Bureau of Standards, Washington, D. C.

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